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THE UNIVERSITY OF ALBERTA

DEVELOPMENT OF AN OIL SANDS HEAT TRANSFER CELL

by



DOUGLAS A. LILICO

A THESIS

SUBMITTED TO THE FACULTY OF GRADUATE STUDIES AND RESEARCH
IN PARTIAL FULFILLMENT OF THE REQUIREMENTS FOR THE DEGREE
OF MASTER OF SCIENCE

DEPARTMENT OF MECHANICAL ENGINEERING

EDMONTON, ALBERTA

FALL 1986

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ISBN 0-315-37853-0

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TITLE OF THESIS

Development of an Oil Sands
Heat Transfer Cell

DEGREE

Master of Science

YEAR THIS DEGREE WAS GRANTED

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THE UNIVERSITY OF ALBERTA
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The undersigned certify that they have read, and recommend to the Faculty of Graduate Studies and Research, for acceptance, a thesis entitled DEVELOPMENT OF AN OIL SANDS HEAT TRANSFER CELL submitted by DOUGLAS A. LILICO in partial fulfillment of the requirements for the degree of MASTER OF SCIENCE.

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Date *Oct 10, 1986*

ABSTRACT

Most of the vast Alberta oil sand deposits are too deep to be strip-mined and must be exploited by in situ methods. As heat transfer is a very important component of in situ production, a study of some aspects of in situ heat transfer in oil sands was initiated by the Alberta Research Council. Implicit in this study was a need for fundamental thermophysical property measurement and detection of heat transfer modes in the oil sands.

An apparatus for the measurement of the effective thermal conductivity of, and the onset of natural convection in, oil sand material under in situ conditions was designed, built and tested. This heat transfer cell measures the one-dimensional steady state heat flux and temperature drop across a sample of oil sands 460 mm in diameter and 40 mm to 300 mm thick. Heat flux is found by measuring the temperature drop across a known thickness of borosilicate glass for which the thermal conductivity is known. Thermal conductivity is measured by heating from above and cooling from below, thus suppressing convection. Natural convection heat transfer effects are measured by heating from below and cooling from above, thereby enhancing convection. Natural convection testing also demands a thicker sample section so a variable thickness capability was built into the cell.

The heat transfer cell measures effective thermal conductivity to an absolute accuracy of 7.2%. Maximum spatial temperature variations within the measurement area are less than $\pm 0.33^{\circ}\text{C}$ while the temperature variations with time when at steady state conditions are less than $\pm 0.01^{\circ}\text{C}$. The effective thermal conductivity of a simulated oil sand was found to be 1.3 W/mK and that of Ottawa sand to be 0.7 W/mK, both of which agree with published values.

ACKNOWLEDGEMENT

I would first of all like to express my appreciation to the Alberta Research Council and the the Alberta Oil Sands Technology and Research Authority for funding and support throughout this study.

I am most grateful to my academic supervisor, Dr. J.D. Dale, and to my project manager, Dr. V.R. Puttagunta, for their invaluable advice, availability and support.

Thanks are also extended to the machinists and technologists at the Oil Sands Research Department of the Alberta Research Council, in particular to Eddie Jossy, Alvin Ewanchuk and Joe Berlingo.

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1. INTRODUCTION

The reducing supply of oil producible by conventional methods is attracting attention to reserves of high viscosity oil contained in oil sand and oil shale deposits. The Alberta oil sand deposits are vast and estimated to contain $2.15 \times 10^{11} \text{ m}^3$ of bitumen in place (Takamura, 1982). Present commercial exploitation is by strip-mining of surface or near surface deposits. However, most deposits are too deep to be economically strip-mined and pilot plants are being used to test in situ production from deeper deposits. The two most popular in situ recovery techniques, steam injection and in situ combustion, mainly depend on preheating the oil sands to increase the mobility of the oil in the formation. The increased mobility allows production to occur, while without heating there would not be any significant oil recovery. Thus, heat transfer in oil sands is of critical importance to the production of oil from deep oil sands deposits.

1.1 Heat Transfer in Oil Sands

The microscopic structure of Alberta oil sands is quite complex (Takamura, 1982) and has four major components: water, bitumen, sand grains, and fines, as shown in Figure 1.1. The sand grains are completely covered by a thin (approx. 10 nm) layer of water, with pendular rings of water surrounding the contact points

Figure 1.1: SCHEMATIC DIAGRAM OF ATHABASCA OIL SAND. has been removed because of the unavailability of copyright permission. Figure 1.1 was a schematic depiction of the microstructure of Athabasca oil sand and included an inset of the thin (10nm) water layer covering the sand grains. The diagram can be found in "Microscopic Structure of Athabasca Oil Sand," by K. Takamura, Canadian Journal of Chemical Engineering, Vol. 60, 1982, pp. 538-545.

between sand grains. Fines are either precipitated onto the sand grain surfaces or are located in clusters which are completely water-filled. The bitumen in the oil sand occupies the remaining pore spaces. Gases present are usually dissolved into the water and bitumen but can form a separate phase at high concentrations. Heat transfer through porous material utilizes three paths (Kunii, Smith, 1960):

1. Through void spaces and fluids by conduction, natural convection, forced convection and radiation.
2. Through a series path composed of both the solid phase and the void space fluids.
3. Through the solid phase alone, with energy being transferred from one particle to the next through the contact area.

The extent to which each path influences the overall heat transfer process depends on the microstructure of the porous material, the various material properties, and the environment the porous material is subjected to, i.e. temperature, fluid pressure and overburden pressure.

Heat transfer through the fluids in the pore spaces is primarily by conduction, natural convection, and forced convection. Heat

transfer by radiation is negligible because of relatively low temperature differences and pore space fluids being primarily liquids. The large number of in situ oil production tests, whether pilot plant or laboratory-based, have resulted in considerable knowledge of forced convection in oil sands (Ward and Ward, 1984); thus it will not be discussed further. Natural convection in the pore spaces can be divided into macroscale and microscale convection. Macroscale convection is the movement of pore fluids throughout the oil sand matrix under the influence of density and concentration gradients. Microscale convection is the movement of pore fluids within a single pore space under the influence of density and concentration gradients.

Heat transfer through the sand grains is by conduction. Heat is transferred by various mechanisms from sand grain to sand grain. These are: conduction through the contact areas between sand grains,

conduction through the pendular water rings surrounding the contact areas, and conduction and natural convection through the pore space fluids.

Heat transfer through the oil sand microstructure can be seen to be very complex; however, the overall heat transfer rate through the oil sands can be compared with the macroscopic temperature gradient to yield an effective thermal conductivity. This effective thermal conductivity would include parallel and series

conduction, microscale convection, and small radiation terms but should not include forced or macroscale convection terms, because these are not present under all-conditions of heat transfer. The effective thermal conductivity of an oil sand is best predicted by empirical equations based on experiments performed on similar oil sands.

1.2 Heat Transfer Cell Aims

The Alberta Research Council's heat transfer project was intended to study the significant areas of heat transfer in oil sands that are not being covered by production studies. This limited the study area to effective thermal conductivity and macroscale convection.

Much of the effective thermal conductivity data published for oil sands lack reliable correlations to more easily measured properties and have been taken at low pressures and moderate temperature. There have been no studies published on macroscale natural convection in oil sands or oil sand-like systems, whether on an experimental or an analytic basis. Hence, it was decided to construct a heat transfer cell or cells that would be able to:

1. Measure the thermal conductivity of oil sands or reconstituted oil sands at simulated subsurface conditions of high temperature, high overburden pressure

and high pore fluid pressure.

2. Aid in the development of a correlation that would allow thermal conductivity to be easily and accurately calculated from more easily measured oil sand parameters.
3. Be able to study macroscale natural convection in an oil sand-like system.
4. Be able to study macroscale natural convection aided by vapourization and condensation in oil sands (the heat pipe effect).

These four aims could be satisfied by either using two test cells, one for thermal conductivity and the other for convection, or by using a single cell capable of measuring both thermal conductivity and convection. The disadvantage of using a necessarily large and cumbersome convection apparatus for thermal conductivity experiments was judged to be more than offset by the advantage of having only one design, assembly and commissioning stage.

This study was limited to the design and initial testing of an oil sands heat transfer cell. The initial testing was limited to thermal conductivity measurements only, but the design should be capable of being used to study macroscale natural convection in an oil sand-like system under similar conditions.

2. LITERATURE SURVEY

2.1 Thermal Conductivity of Oil Sands

Previous studies of oil sand thermal conductivity have primarily been experimental. Both steady state and transient methods of measuring thermal conductivity have been used, as have a wide spectrum of operating conditions. Short summaries of the most significant of these studies follow, in chronological order, and Table 2.1 provides a summary of the experimental conditions and results obtained for those studying the thermal conductivity of Alberta oil sands.

K.A. Clark (1944) published thermal conductivity data for both natural oil sands and repacked oil sands. He used a steady state, radial heat flow device in which a heater was located axially in a cylinder of oil sand and the steady state radial temperatures were noted. The radial temperature drop and the power consumed by the heater gave the thermal conductivity. The average temperature was 45°C, the pressure was atmospheric and there was no pore fluid control. Clark calculated a thermal conductivity of 1.5 W/mK for the undisturbed samples and 1.3 W/mK for remoulded oil sands with the same oil content. He also found that the thermal conductivity decreased as the bitumen content decreased.

W. Woodside and J.M. Messmer (1961) examined several experimental techniques and selected the transient, heated line source method. The reasons for selecting this method in preference to steady state methods were that steady state methods require complicated guarding systems to minimize edge or end effects, and if the porous system was only partially saturated with liquid the long term temperature gradients of the steady state apparatus could result in non-uniform saturation due to osmosis. Woodside and Messmer used the transient probe method on gas- and water-saturated sand where the power consumed by a line heat source was compared to the temperature rise in the sand matrix surrounding the heater. The advantages of this method are its high test speed and therefore reduced saturation redistribution. However, there are two problems with the technique: contact resistance between the heater and sand matrix causing possible large errors, and large temperature gradients near the heater causing some saturation redistribution problems.

W.M. Somerton, J.A. Keese and S.L. Chu (1974) determined the thermal conductivities of Ottawa sand, Del Monte sand, and Kern River oil sands saturated with air, brine, and solvent. They used a steady state axial heat flow device that measured heat flux by measuring the temperature drops across materials of known thermal conductivity. The tests were conducted with an overburden pressure of 2.4 mPa, a pore fluid gauge pressure of about 17 kPa, and a temperature of 52°C. They found the effects

of overburden pressure to be small.

A.P. Deston (1976) reviewed the literature available on the effect of pressure on the thermal conductivity of oil sands and concluded that pressure has its greatest effect through liquid saturation and porosity, and that the direct effect of pressure on thermal conductivity is quite small, on the order of 0.2% per MPa pressure change.

W.R. Lindberg (1980) published details of a study of the thermal conductivity of Utah oil sands where he used a steady state axial heat flow device. He examined various steady state methods and used a technique similar to Somerton, Keese and Chu. The tests were conducted at 40°C to 120°C with an unreported overburden pressure and at atmospheric pore fluid pressure.

The thermal conductivities of reconstituted Athabasca oil sand samples were measured by M.R. Cervenán, F.E. Vermeulen and F.S. Chute (1981). They used three different apparatuses: hot plate, co-axial and transient probe. The hot plate apparatus was a steady state axial heat flow device where heat fluxes were determined by measuring the power consumed by the heater. The thermal conductivity was determined by measuring axial temperature drops within the oil sands. The co-axial apparatus was a steady state radial heat flow device that used an inner cylinder as a heater. This method gave results similar to those

obtained by the hot plate method. The transient probe had large contact resistance problems but gave results similar to the other apparatuses when coated with silicone grease. The tests were done at room temperature and atmospheric pressure and gave thermal conductivities of 1.3 W/mK to 2.0 W/mK as the water saturation varied from 0.9% to 44%.

G.A. Karim and A. Hanafi (1981) studied oil sand thermal conductivity using repacked samples in a steady state radial heat flow apparatus similar in theoretical basis to that used by K.A. Clark (1944). The tests were conducted at atmospheric pressure with a temperature range from 20°C to 120°C. Thermal conductivity was found to be about 1.5 W/mK and to decrease with decreasing bitumen content or increasing temperature. Water saturation values were not noted.

N. Seki, K.C. Cheng and S. Fukusako (1983) studied oil sand thermal conductivity using a transient radial device. Repacked samples were heated from the outside and their axis temperature measured. The tests were done from 15°C to 480°C with no overburden pressure and no pore fluid control. Thermal conductivity values ranged from 1.07 W/mK to 0.35 W/mK with a major perturbation at 100°C, probably because the water in the sample boiled off.

J.D. Scott and A.C. Seto (1985) studied oil sand thermal

Table 2.1: Alberta Oil Sands Thermal Conductivity Studies

Date	Investigator	Temperature (°C)	Pore Pressure	Overburden Pressure	Thermal Conductivity (W/mK)	Comments
1944	Clark	45	Atmospheric	Atmospheric	1.5	Natural
		45	Atmospheric	Atmospheric	1.3	Repacked
1981	Cervenán et al.	25	Atmospheric	Atmospheric	1.3-2.0	Repacked
1981	Karim, Hanafi	20-120	Atmospheric	Atmospheric	0.6-1.2	Repacked
1983	Seki et al.	15-480	Atmospheric	Atmospheric	0.35-1.1	Vapourization
1985	Scott, Seto	20-150	2 MPa	10 MPa	1.7-2.0	Natural

conductivity using undisturbed samples in a transient radial apparatus. The apparatus used a central heater and thermocouples which measured radial temperature distribution. The temperature range was 20°C to 150°C, an overburden pressure of up to 10 MPa was applied and pore fluid pressures of 2 MPa were used. The high pore fluid pressure negated the problem of thermal contact resistance. The tests revealed thermal conductivity values varying from 2 W/mK to 1.7 W/mK as the temperature increased. Additional results using this apparatus can be found in Seto (1985).

2.2 Multifluid Natural Convection in Porous Media

Macroscale natural convection in oil sands is the movement of pore fluids, and hence energy, in the oil sand layer due to buoyancy effects. Oil sand pore fluids are not single component and therefore multicomponent natural convection in porous media was studied.

Multicomponent convection can be divided into multiphase convection of a single fluid (e.g. boiling of water), convection due to concentration and temperature differences (e.g. thermohaline convection in geothermal systems) and multifluid convection. Multifluid convection is the type most applicable to oil sands because they contain both water and bitumen. When the pore fluid pressure and temperature distribution allow

vaporization and condensation of the bitumen light ends or water. multifluid convection is enhanced. This is called the heat pipe effect.

The convection of fluids in a horizontal layer of a porous medium can best be characterized by the Rayleigh number (Ra), which is the ratio of the buoyant to viscous forces (Katto, Masuoka, 1967) as follows:

$$Ra = \frac{g\beta \Delta T k \delta}{\alpha_m \nu} \quad 2.2.1$$

where:

g = local gravity acceleration constant (m/s^2)

β = coefficient of volume expansion of the fluid ($1/K$)

ΔT = temperature difference across the layer ($^{\circ}C$)

k = permeability of the fluid-saturated porous medium (m^2)

δ = thickness of the layer (m)

ν = kinematic viscosity of the fluid (m^2/s)

and

α_m = thermal diffusivity of the medium (m^2/s)

$$\alpha_m = \frac{\lambda_m}{(\rho C_p)_f} \quad 2.2.2$$

where:

λ_m = thermal conductivity of the saturated porous medium
(W/mK)

ρ_f = density of the fluid (kg/m^3)

C_{p_f} = specific heat capacity of the fluid (J/kg K)

When a fluid-saturated horizontal layer of a porous medium is heated from below, convection does not occur until the buoyant forces overcome the viscous forces. Thus, until the Rayleigh number reaches a critical limit (Ra_c) no convection occurs. When Ra_c is exceeded, convection occurs and as Ra continues to increase the heat transfer through the medium increases and the pattern of fluid motion can change. The value of Ra_c and the patterns of fluid motion are dependent on the fluid properties and the shape of the layer, but for a single, constant viscosity fluid, in an infinite layer Lapwood (1948) showed that $Ra_c = 4\pi^2$.

Two papers have been published on multifluid convection in porous media. M.L. Lawson and W.J. Yang (1975) published an experimental study of the onset of convection of a He-N₂ mixture in a porous matrix of steel ball bearings. The critical Rayleigh number was determined by using the Schmidt-Milverton principle (a sudden increase in the effective thermal conductivity) in an axial steady state device using an asbestos disc as a heat flux meter. They found that Ra_c for a pure gas is lowered by the

presence of another gas and has a minimum value at a certain composition of the binary mixture. Also, M. Brand and V. Steinberg (1983) conducted analytical studies of miscible liquid mixtures to find criteria for the onset of convection. The liquid mixtures studied were water-ethanol, ethanol-toluene, and ^3He - ^4He . The critical Rayleigh number calculated from their results for 60% water-40% ethanol was 29.4.

3. HEAT TRANSFER CELL DESIGN

3.1 Selection of Cell Type

An apparatus used for the investigation of natural convection has a more stringent set of criteria than a thermal conductivity measuring device. Therefore, the heat transfer cell was selected to be a convection device that could be easily modified to measure thermal conductivity.

Convection in an oil sand system can be studied using either a horizontal or vertical temperature gradient. Systems under a horizontal temperature gradient convect very easily, but the equations used to describe the fluid motion and heat and mass transfer are very complex and it would be difficult to separate the specific effects of the oil sand system. On the other hand, it is harder to initiate convection in systems with a vertical temperature gradient, but the onset of convection in these systems is a thoroughly studied phenomenon and specific effects of oil sand can be detected. The Schmidt-Milverton principle (Lawson, Yang, 1975) for detecting the onset of convection is essentially based on the measurement of the system's steady state effective thermal conductivity, with convection beginning when the effective conductivity suddenly increases. An apparatus designed for detecting the onset of convection using this principle would also be able to measure oil sand effective

thermal conductivity, if convection could be suppressed.

A Schmidt-Milverton apparatus is used to measure the uniaxial steady state thermal conductivity of a layer of sample which is heated from below and cooled from above. However, in a convection-suppressed thermal conductivity apparatus the sample is cooled from below and heated from above. Therefore, a cell containing a horizontal layer of sample that can be heated or cooled from both top and bottom would satisfy both intended areas of investigation. Also, an apparatus intended for convection studies needs a thicker layer of sample than for thermal conductivity studies; thus, for both studies to be done on one apparatus variable sample thickness is necessary and important.

Steady state effective thermal conductivity can be measured by comparing the uniaxial heat flux through a sample with the uniaxial temperature gradient across the sample. The temperature gradient is usually approximated by measuring the temperature drop across a known thickness of sample. The heat flux for a lower thermal conductivity material can be measured by using the hot plate method (ASTM, 1976a) or by the comparative heat flux meter method (ASTM, 1976b). R.P. Tye (1968) published a study of the various methods of measuring thermal conductivity, including information on methods of measuring heat fluxes. The guarded hot plate method is a primary method that directly measures heat fluxes and is the most suitable for a low to medium range of thermal

conductivities. In this method the power consumption of a main heater is measured to give the heat flux. Usually two samples straddle the main heater with cooling heat sinks outside the samples. Edge losses must be negligible in order to find the heat flux in the hot plate method but the comparative heat flux method is slightly more forgiving of edge effects. The comparative heat flux meter method is a secondary method in which heat fluxes are calculated from temperature drops through materials of known thermal conductivity. This method usually involves sandwiching the sample between two heat flux meters and is suitable for a medium range of thermal conductivities. The accuracy of this method is limited by the accuracy of available thermal conductivity data for the heat flux meter material.

For the planned large size of the heat transfer cell, the construction necessary for the in situ operating conditions, and the two very different operating modes, the accurate measurement of power delivered to the sample would be very difficult. Therefore, a comparative heat flux meter method was selected as it could measure the heat flux through the sample under difficult conditions.

3.2 Operating Conditions

The design operating conditions of the heat transfer cell were selected so as to mimic a real underground situation as closely

as possible. The design overburden pressure range was set at 3.4 to 6.8 MPa to simulate actual overburden pressure and to ensure grain to grain contact. The temperature range was chosen up to 300°C in order to simulate in situ conditions near high temperature steam injection wells and to provide a large temperature difference capability for the convection studies. The pore fluid pressure range was 3.4 to 6.8 MPa which approximates real in situ conditions, ensures that water phase changes do not occur and that there is 100% fluid pore saturation. However, the lighter ends present in the bitumen may not stay in the liquid phase at these pressure and temperature limits. For simulated oil sands a sand pack porosity of about 35%, permeability of 5 darcies, and the sand grain size range of 70 - 140 mesh were chosen in order to approximate actual high-quality oil sands.

3.3 Cell Design

The primary design constraint for the heat transfer cell was the diameter and height of the cylinder containing the sample for convection studies. The same diameter, but thinner, sample would be used for conductivity studies. The cell was designed so that a 5-darcy permeability sand pack filled with water should convect when subjected to a temperature difference of 230°C. If subsequent studies show convection does not occur the sand pack permeability can be reduced. Using the effects of variable water

viscosity (Kassoy, Zebib, 1975) and aspect ratio (Zebib, 1978) it was found that a 454 mm diameter cylinder should need a height of 750 mm in order to convect water in a 5 darcy sand pack. For detailed calculations, see Appendix A.

A diagram of the heat transfer cell in thermal conductivity mode is shown in Figure 3.1, and in convection mode in Figure 3.2. An asbestos-cement (A/C) water transmission pipe rated at 4.8 MPa and with a 460 mm inner diameter was selected for the wall of the cell due to its similar thermal conductivity to oil sand (1 W/mK versus 1.5 W/mK), high temperature durability, and pressure capability. Thus the height of the cell could be varied by simply cutting the pipe to differing lengths. Pyrex 7740 discs 31.8 mm thick and 454 mm in diameter are used for heat flux meters as thermal conductivity values are well-known for this material. Identical heating/cooling heads, as detailed in Figure 3.3, are installed at each end of the pipe. The heads each contain a sandwich of a silicone oil-fed cooling plate, an electrical heating plate, and a copper sealing plate. The copper sealing plate, shown in Figure 3.4, contains temperature control thermocouples, ports for sensor leads and fluid injection, and the sealing surface for the flexible asbestos-metal (Flexitallic) gasket used against the asbestos-cement pipe ends. Each heat flux meter is glued to a copper sealing plate with a silicone rubber pad. A 762 mm diameter steel pipe surrounds the cell and acts as both a temperature guard against radial heat losses and

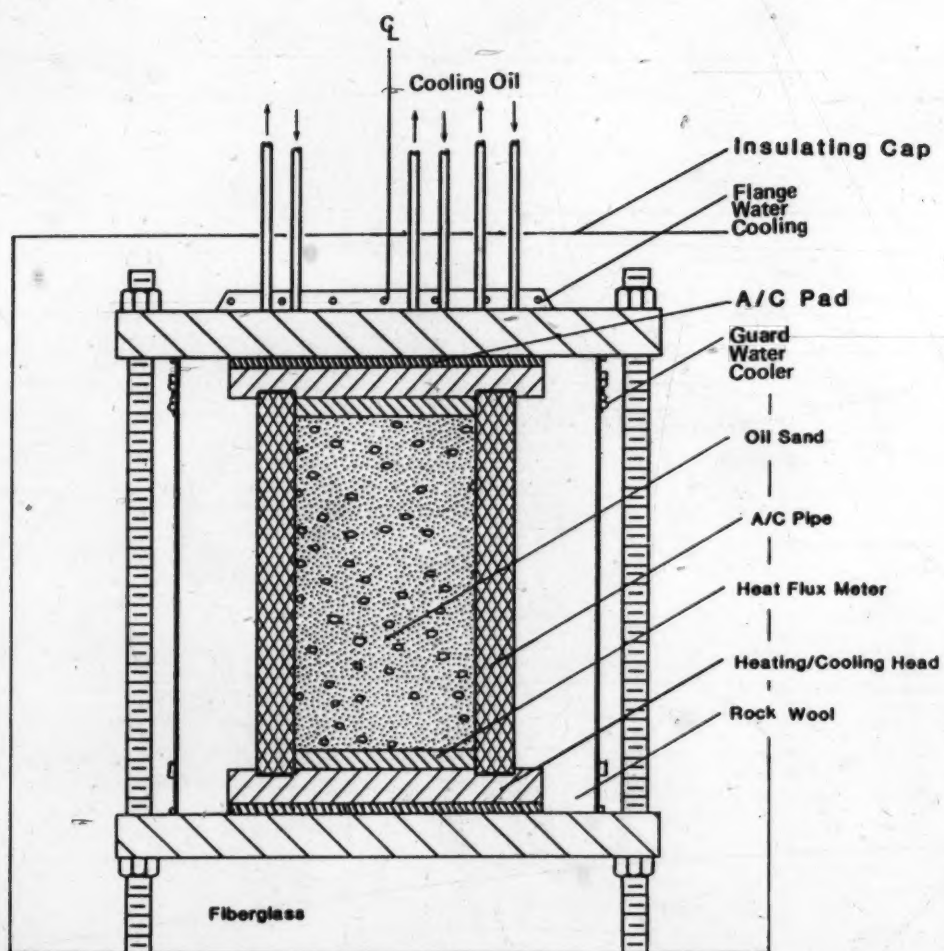


FIGURE 3.2 NATURAL CONVECTION MODE OF HEAT TRANSFER CELL

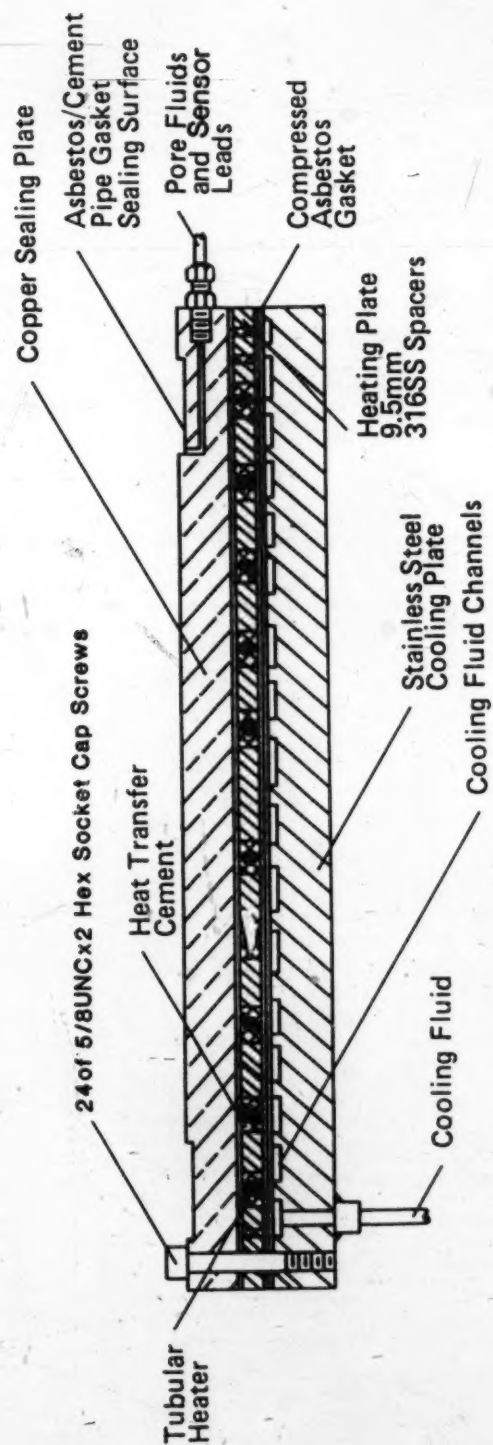


FIGURE 3.3 HEATING/COOLING HEAD SECTION

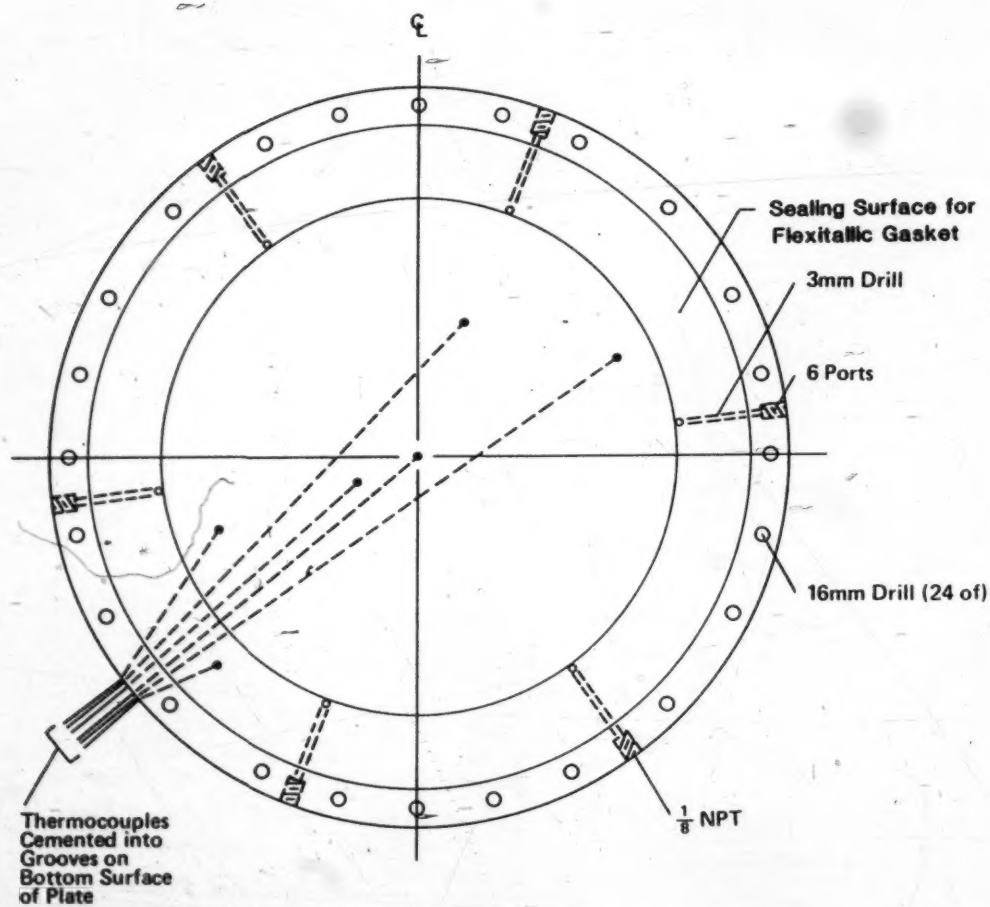


FIGURE 3.4 COPPER SEALING PLATE

as protection against pore fluids if the asbestos-cement pipe fails. Heaters are located on the top and bottom of the guard and the cold end also contains a water-fed cooling coil. The cell assembly is compressed by two .24" 600-pound flanges which use 12 studs to axially load the system. The flanges, which contain auxiliary coolers, also have welded-on rings to contain the steel pipe guard. Asbestos-cement pads are used between the flanges and the heating/cooling heads as high strength thermal insulation. Rock wool insulation is installed between the cell walls and the guard, and an insulating cap of fiberglass surrounds the entire assembly.

There are three groups of heaters: the main heating plates, the guard-heaters, and the pore fluid control system heaters. The main heater plates, as shown in Figure 3.5, are two identical 635 mm diameter, 12 mm thick discs that contain coiled tubular heaters embedded in Thermon heat transfer cement and enclosed in stainless steel. Stainless steel spaces fill the areas between heaters and provide the required compressive strength. There are two separate circuits inside each heating plate, one controlling the inner 381 mm diameter and the other the outer annulus. The heaters are run by computer-controlled power controllers and operate at either 103 VAC or 260 VAC. On 260 VAC the maximum total power consumption of both main heating plates is 15 kW.

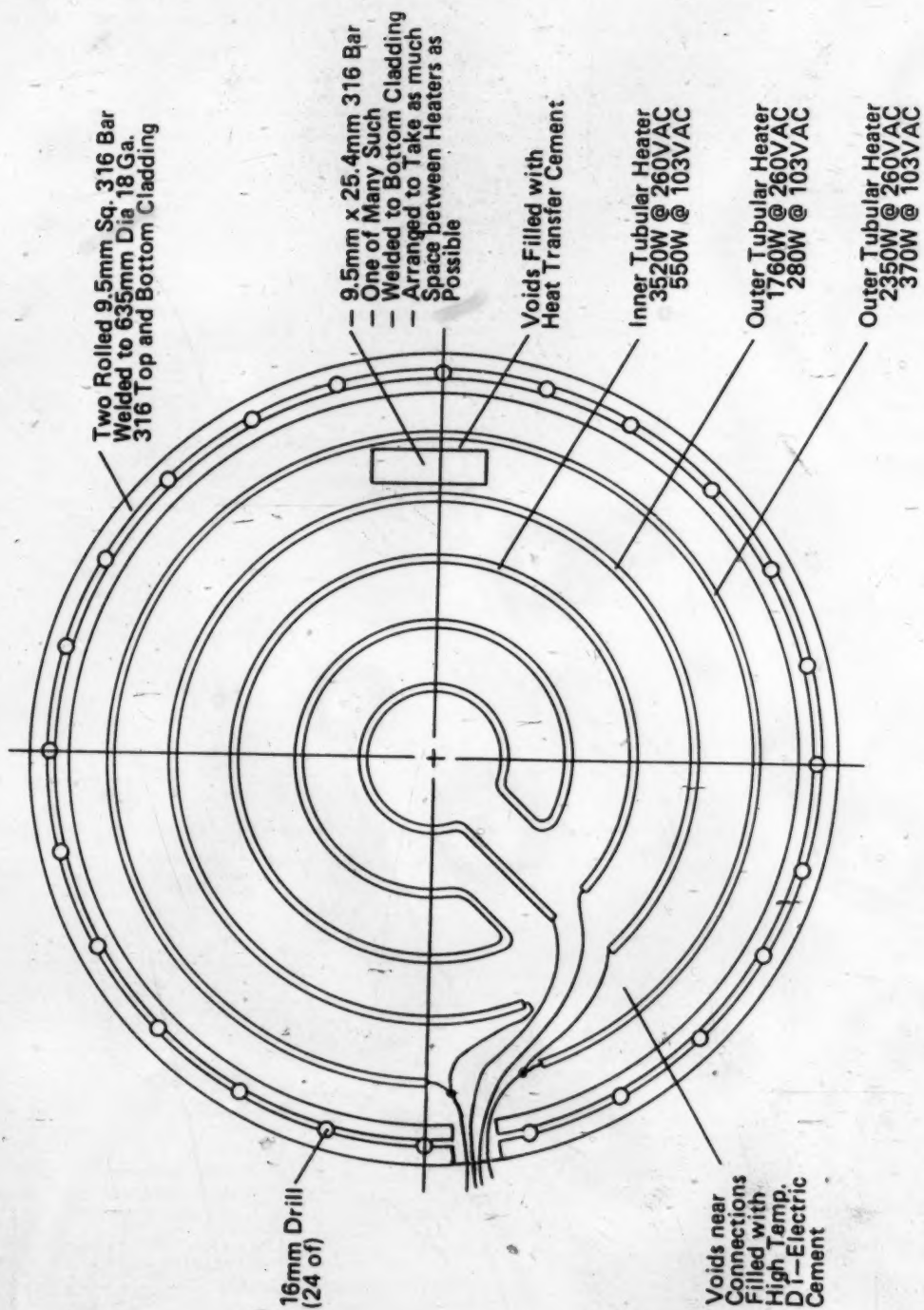


FIGURE 3.5 HEATING PLATE

The guard heaters are heavily-insulated heat tapes, two on the hot end of the guard and two on the cold end. These run on 120 VAC and consume up to 620 watts each. Control is accomplished with two solid state temperature controllers, one for each end of the guard. The pore fluid control system heaters are 120 VAC heat tapes controlled by a single temperature controller.

The cell cooling circuits are shown in Figure 3.6. Two cooling fluids are used, Dow Corning 210, a high temperature silicone heat transfer oil and water. The oil is pumped and kept at a constant temperature by a Haake N-3 circulator, and is run through one of the two 25 mm thick stainless steel cooling plates, shown in Figure 3.7, contained in the heating/cooling heads. Cooling is normally used on the cold end of the cell. The cooling plates have three parallel counter-current paths inside them. Water is used to cool the cold flange for near room temperature testing and to cool the low temperature end of the guard pipe. The water coolers are made of 6.35 mm diameter copper tubing wound onto the guard or spiraled onto the outer surface of the flange and then embedded in Thermon heat transfer cement.

A schematic diagram of the pore fluid control system is shown in Figure 3.8. Pore fluids are contained in an expansion tank which drains through a manifold to six injection ports in each of the copper sealing plates contained in the heating/cooling heads. Pore pressure is maintained by high pressure nitrogen gas acting

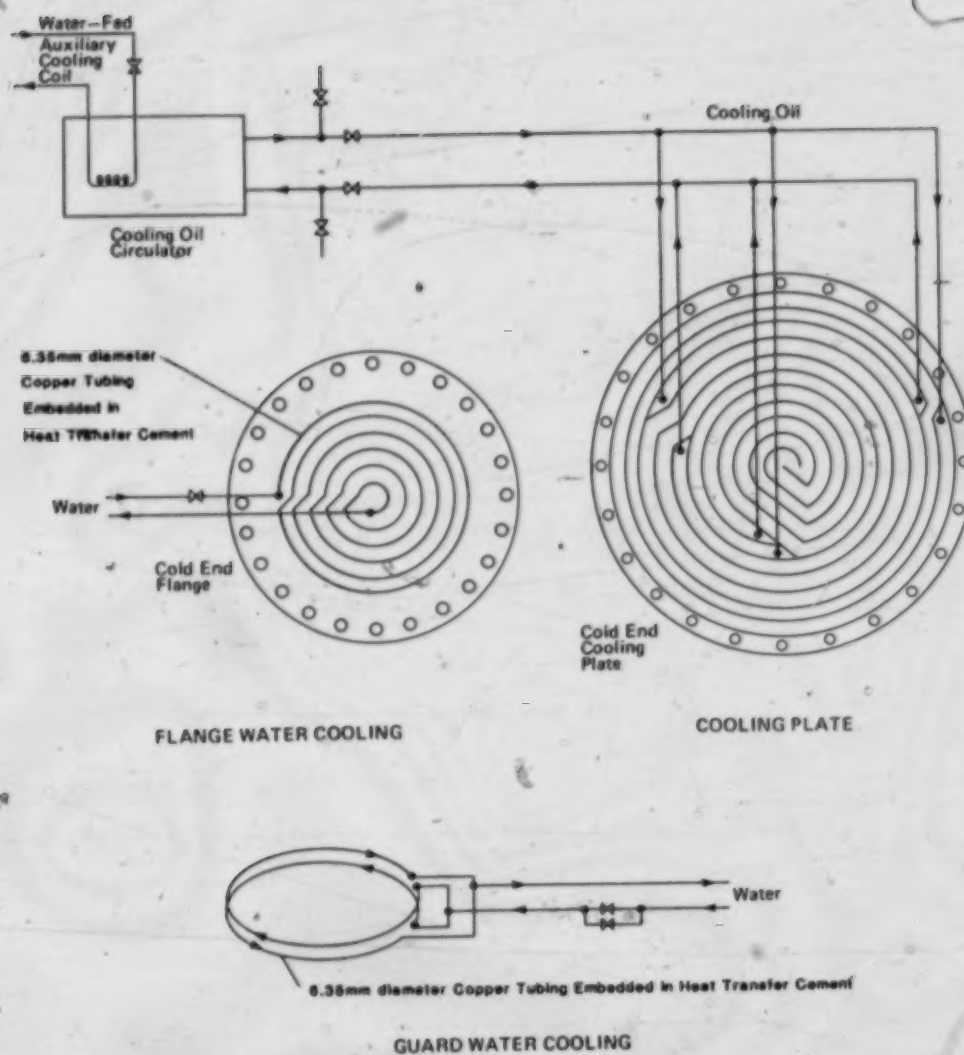


FIGURE 3.6 COOLING CIRCUITS

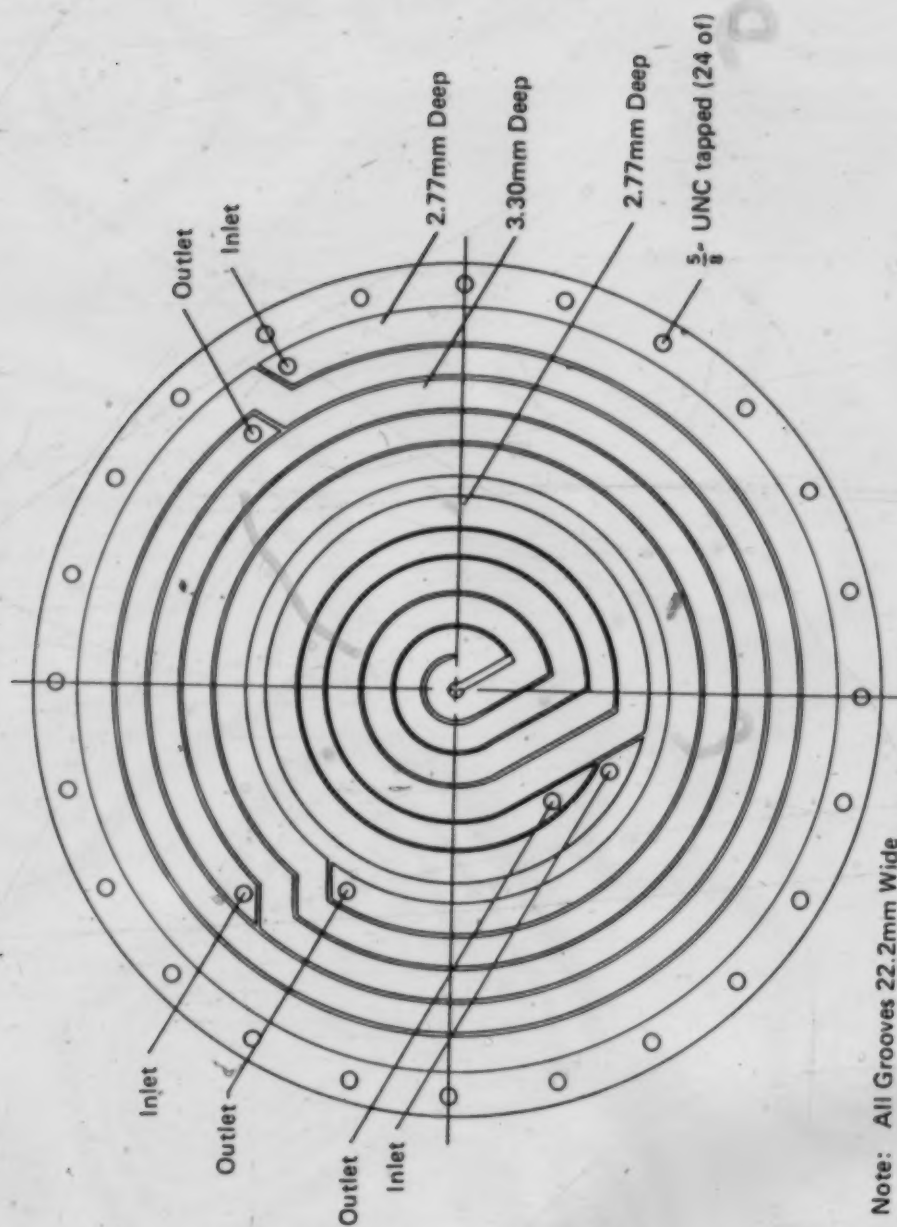


FIGURE 3.7 COOLING PLATE

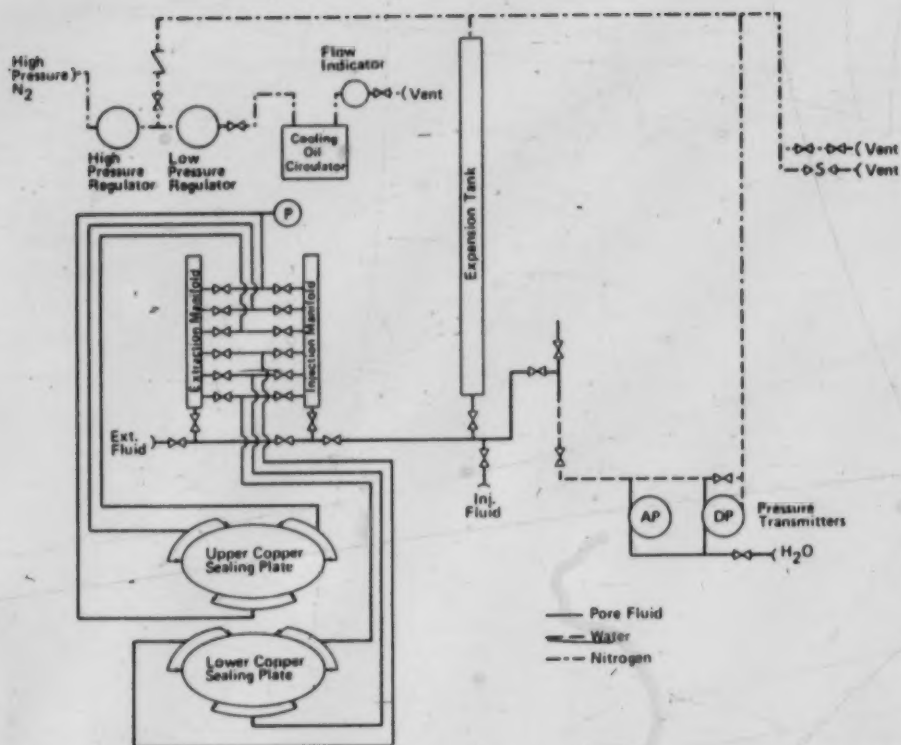


FIGURE 3.8 PORE FLUID CONTROL SYSTEM

on the expansion tank pore fluids. The head of pore fluid inside the expansion tank is measured by a differential pressure transmitter and the pore fluid pressure is measured by an absolute pressure transmitter. A low pressure nitrogen blanket is maintained over the circulator in order to reduce oil oxidation at high temperatures.

The detailed calculations of the heat transfer rates through the cell for heater sizing, of the thermal expansion effects and mechanical strengths are presented in Appendices B, C and D.

3.4 Cell Monitoring and Control

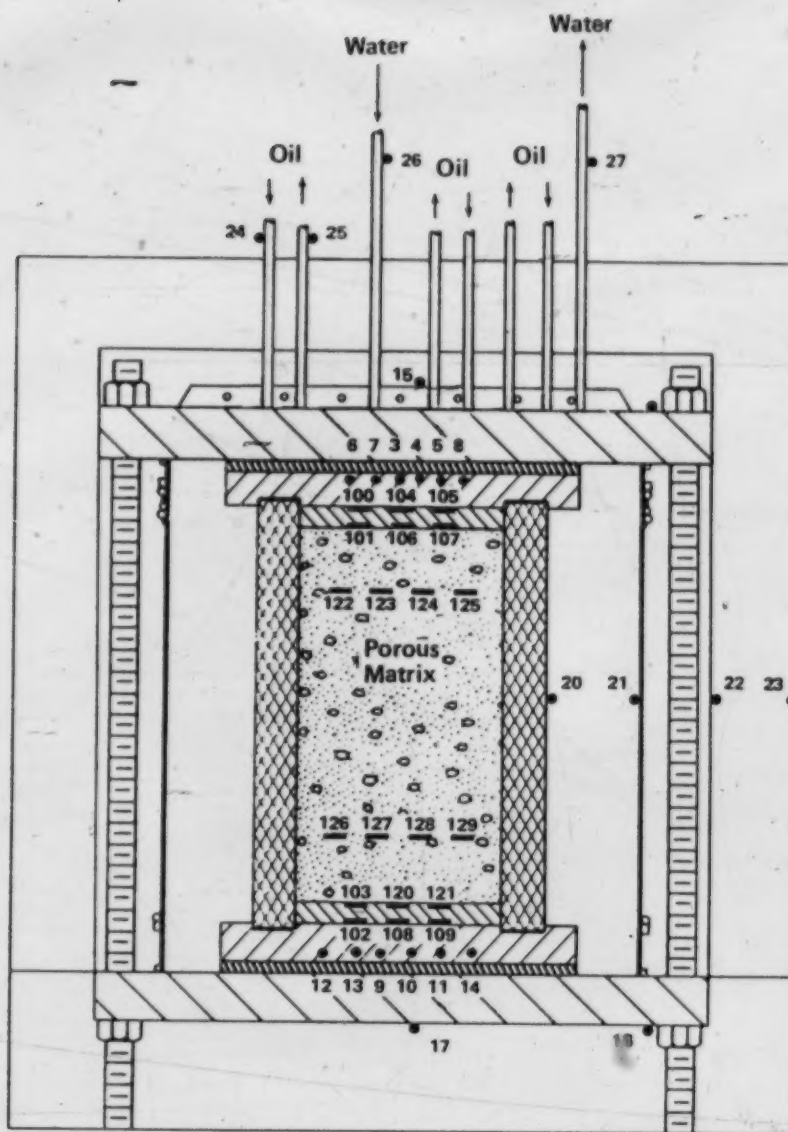
Monitoring and control of the heat transfer cell is handled by a Hewlett-Packard 3054A data acquisition and control system with an uninterruptable power supply. The configuration of the HP3054A is a 3456A digit voltmeter with 6 1/2 digit capability and a 3497A multiplexer with a 3498A multiplexer extender. The total capacity of the multiplexers are 15 data acquisition and control cards. The system uses three software thermocouple-compensated 20-channel multiplexer cards, two uncompensated 20-channel multiplexer cards, one 8-channel actuator output card and two 2-channel programmable 0-20 mA current supply cards. The 3054A is controlled by a HP85B computer through an HP-IB bus.

Temperature measurements throughout the cell were made both by E-

type thermocouples monitored using the three software thermocouple-compensated multiplexer cards, and by platinum resistance thermometers (RTDs) monitored using the 4-wire offset compensated technique with the two uncompensated multiplexer cards. A typical layout for the major temperature measurement points is shown in Figure 3.9. RTDs and thermocouples were calibrated both by using a Hewlett-Packard quartz thermometer in a stable ice or oil bath for temperatures up to 150°C, and by using a high purity (ACS certified) tin solidification bath for a 231°C calibration.

Pressure measurements are taken with two Rosemount Alphaline 4000 pressure transmitters powered by a Hammond 24 VDC supply. The output currents of 4-20 mA from the transmitters are converted to 0.2 - 1.0 VDC by two precision 50 ohm resistors and the voltages are read by channels two and three of the first software thermocouple-compensated multiplexer card. Channel one of the first software thermocouple-compensated multiplexer card records the on or off state of the Haake circulator.

The heat flux meters, shown in Figure 3.10, are Pyrex 7740 discs grooved and cut into six equal wedges. RTDs or thermocouples are glued into the grooves with silicone adhesive. The leads for the heat flux meters run through the injection ports in the copper sealing plate and through the injection tubing supplying the ports, and exit the tubing through teflon and silicone pressure



• Thermocouple - E Type
 — RTD - 100 Ω

FIGURE 3.9 TEMPERATURE SENSOR LOCATIONS
 Typical Setup with Channel Numbers

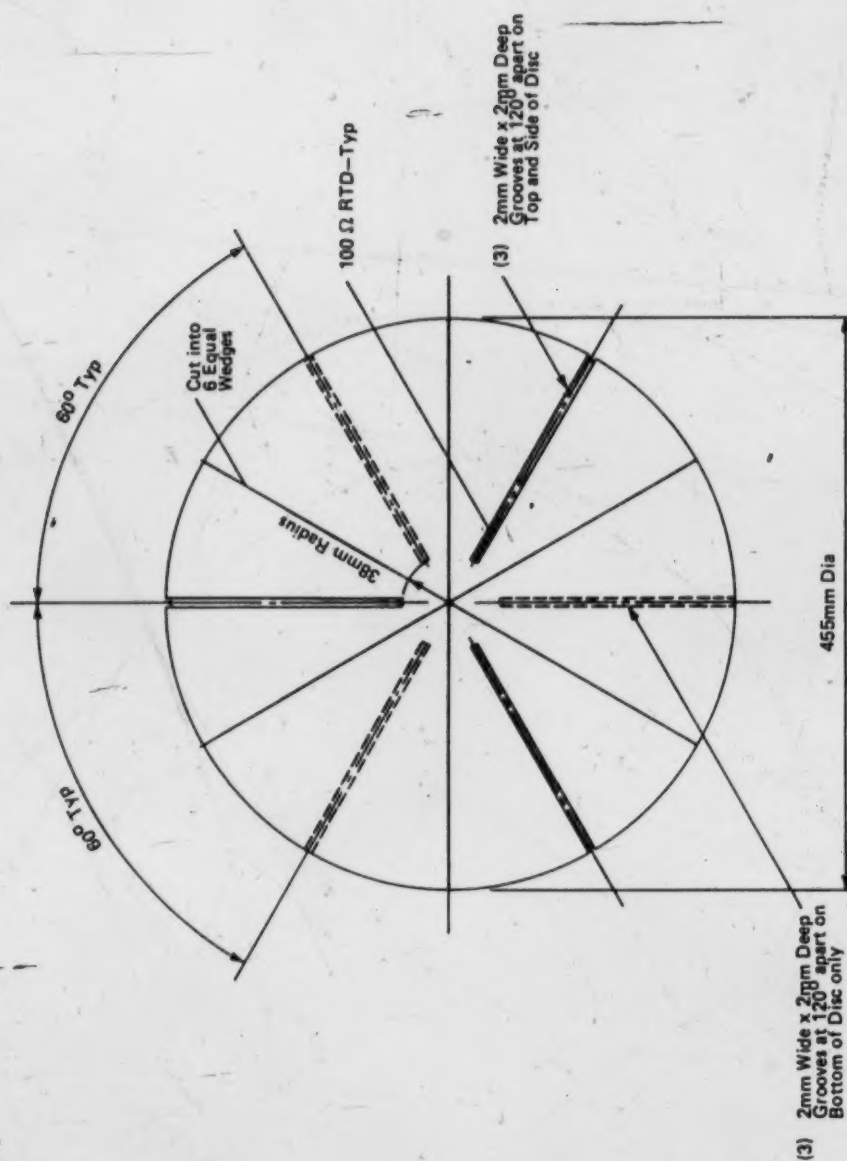


FIGURE 3.10 HEAT FLUX METER

seals.

The probes for monitoring sample temperatures are shown in Figure 3.11 and their arrangement in the cell is shown in Plate 3.1. The probes are either 1.6 mm stainless steel tubing with 100 ohm RTDs at the measurement points, or 3.2 mm stainless steel tubing with E-type thermocouples at the measurement points. The temperature sensor leads were brought through ports in the cell wall, which were sealed by teflon-taped pipe threads in the asbestos-cement.

Two 350 ohm strain gauges are used in the cell to measure overburden and cell wall strain. The resistance of the strain gauges is measured using the same 4-wire offset compensated method as the RTDs. The first gauge monitors lower heat flux meter compression to allow calculation of overburden pressure, and the second is fixed to the asbestos-cement pipe wall to measure wall compression and hence gasket seating.

The monitoring and control program for this system is written in HP Basic and utilizes HP-supplied subroutines wherever possible. The computer system first warns the operator and then aborts the experiment if the pore fluid pressure or differential pressure in the pore fluid expansion tank varies beyond user-selected limits. An experiment is also aborted if the Haake circulator shuts down.

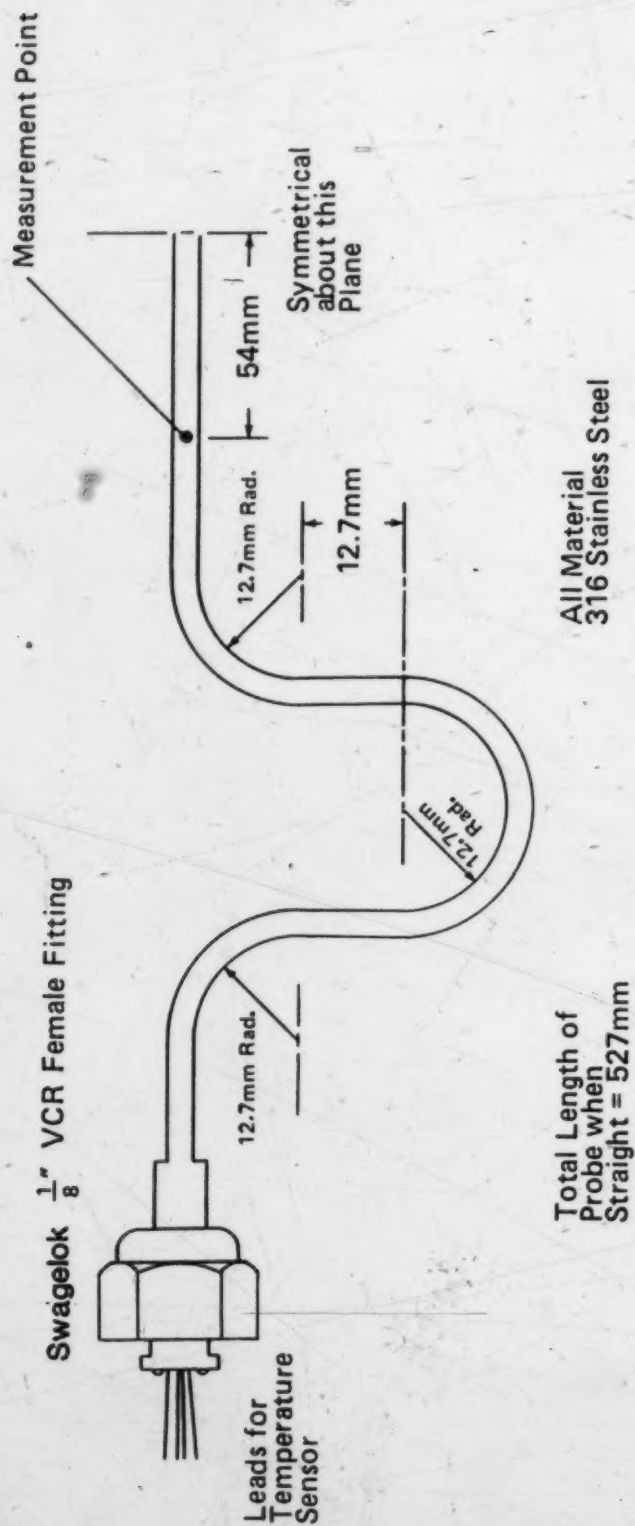


FIGURE 3.11 TEMPERATURE PROBES

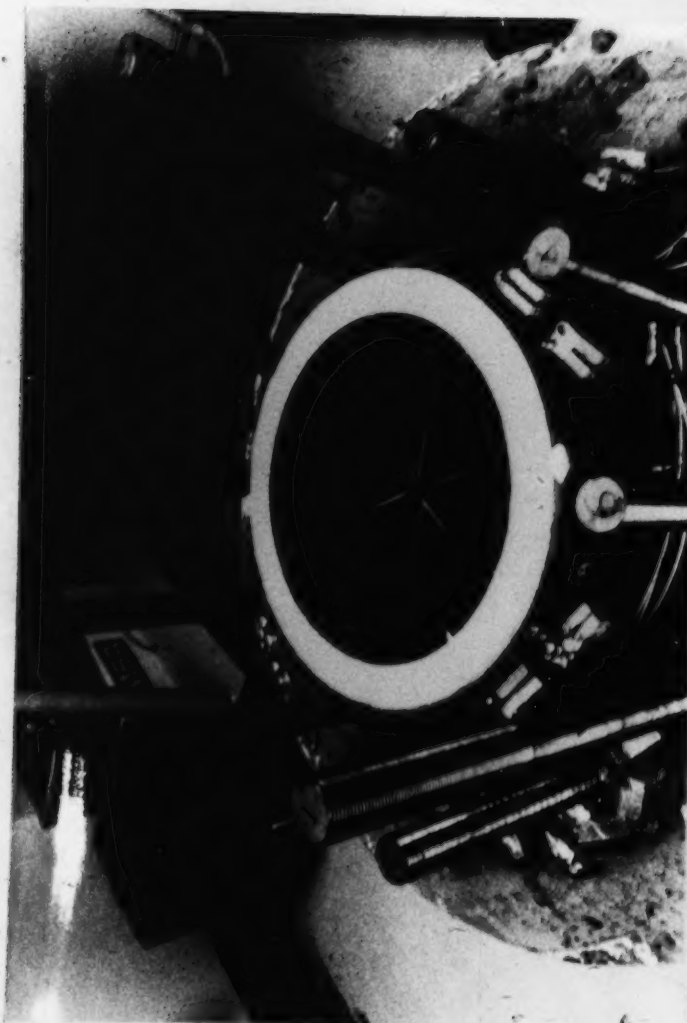


PLATE 3.1 PROBE ARRANGEMENT

if temperature variations across the copper sealing plate exceeded user-selected limits, or if the user requests a shut-down. During normal operation the computer system will read, but not record, only the data necessary for main heater control and abort testing. Upon a status request the system will read and print only the most significant data. Upon a data request the system will read and store on tape all sensor output. A printout of this stored data occurs when an experiment is aborted. The program does not permit sealing plate set point temperatures to be modified while the experiment is underway.

The Haake cooling oil circulator is individually controlled and only reports its on or off condition to the computer. This is accomplished by the closing or opening of an integral relay. When closed the relay causes a 24 VDC reading to be placed on channel 1 of the first software thermocouple-compensated card.

The power system for the cell is shown in Figure 3.12 and uses both the actuator output card to control the system relays, and the current supply cards to control the main heater power controllers. Main heater control is accomplished by PID control and by voltage selection. The PID control has two rates of gain: a high rate for far from the set point, and a very low rate for close to the set point. The rates of gain, as well as the integral and derivative control constants, are user-selectable. The main heaters can be run on either 260 VAC or 103 VAC,

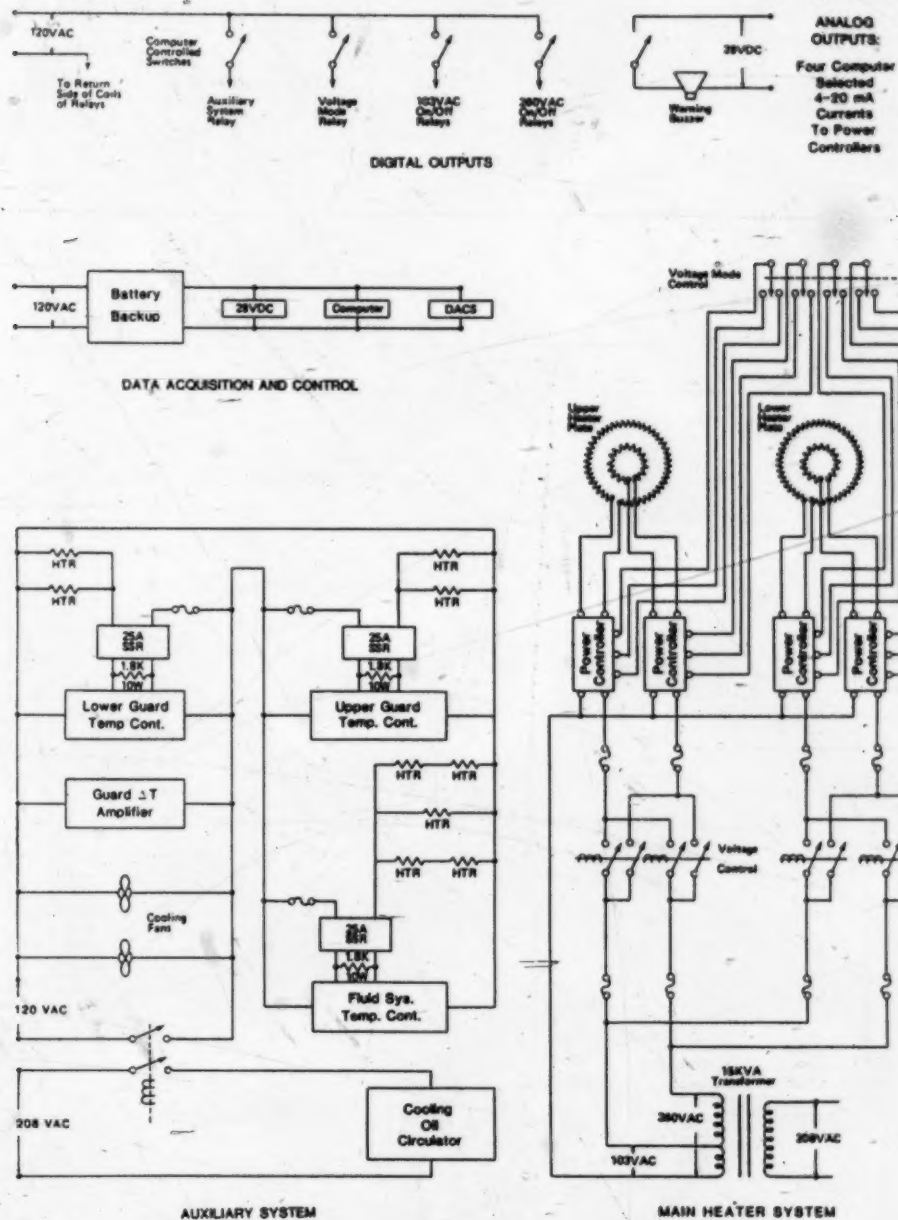


FIGURE 3.12 CELL POWER SYSTEM

depending on user preference, with computer-controlled relays selecting the voltage and setting the power controller for that voltage. Usually 260 VAC was used for fast warmup, and 103 VAC was used for minimum error at steady state conditions.

The guard heaters are controlled by independent temperature controllers. The temperature controllers monitor the radial temperature difference across the insulation between the guard and the cell wall. The difference is measured by a two-point thermopile with the voltage produced being amplified so that a 1°C temperature difference produces a 1 mV signal to the temperature controllers.

The pore fluid control system temperature is maintained by an independent temperature controller handling 5 heat tapes in series combination such that temperatures are $\pm 5^{\circ}\text{C}$ across the system. The pore fluid control system controls pressure by slowly bleeding high pressure nitrogen gas through a gas regulator.

3.5 Operating Procedures

There are two types of samples usable in the heat transfer cell: clean, dry sand and pre-saturated materials. The testing of the cell was done using clean, dry sand only. The procedure for packing dry sand consists of first to position, connect the

sample temperature sensing probes and then packing sand into the sample volume by pouring it through a distributor constructed similarly to that described by Wygal (1962). The distributor uses 5 layers of 16-mesh 304 stainless steel screen spaced 50 mm apart to distribute the sand which then falls 350 mm into the cell through a 500 mm diameter steel tube. The mesh layers and the tube are grounded to reduce static electricity effects. The resulting porosity with 70-140 mesh Ottawa sand is 36%. The proposed procedure for packing pre-saturated materials is to tamp them into the cell which has the heat flux meters replaced with temporary steel spacers that can more easily absorb the shock of tamping. Once the sample is tamped, the assembly is frozen, and then the steel spacers are removed. This method has been successfully used at the Alberta Research Council by R. Ridley and others to prepare laboratory scale oil sandpacks for steam flooding experiments. When sufficient material is built up grooves are to be cut and the probes placed in them. The heat flux meters are to be replaced with temporary steel spacers that can more easily absorb the shock of tamping. The sample is packed to a height such that when the cell is compressed to seat the gaskets, the correct amount of overburden pressure was also applied. Some variation from the necessary sample thickness, approximately 2-3 mm, is allowable as the gaskets can withstand some over-compression and the silicone pads under the heat flux meter are highly compressible.

Once the sample is in place, the upper heating/cooling head with gasket is placed on top of the cell. The guard is then put on, has its cooler connected, the space between the cell wall and guard tightly packed with rock wool insulation, and the radial temperature sensors set in place. It should be noted that at every step of the assembly process, the sensors in every new component attached are connected and tested. Then the asbestos-cement pad is lowered onto the upper heating/cooling head, followed by the upper flange. The flange is tightened using six Holmatro hydraulic stud tensioning jacks. The jacks are placed on one set of six studs (every other stud) and the correct hydraulic pressure for the required stud stress is applied. The stud nuts are then brought into contact with the flange and the jacks removed and placed onto the other set of six studs. This process is repeated two to three times in order to very accurately and evenly tighten the studs so as to not damage the heat flux meters. The normal necessary hydraulic pressure is 31 MPa for a stud stress of 145 MPa and a gasket sealing stress of 62 MPa. Once the flanges are tightened, the cell is pressure tested for leaks. If no leaks are found, fiberglass batts are packed around the studs and then a large fiberglass insulation cap of 150 mm thickness is placed over the cell.

If the sample in the cell is dry sand, saturation of the pack is done by a vacuum method or a flow method. Vacuum saturation

involves pulling a vacuum on the extraction fluid port (see Figure 3.8) and injecting fluid at the injection fluid port. Flow saturation involves flowing two to three pore volumes of fluid through the cell by connecting into the six injection tubes. The fluid is then injected and extracted from different ports in order to thoroughly sweep the cell. The mass of the extracted fluid and the volume of the injected fluid is measured in order to do a mass balance to find the residual saturation in the cell sample.

Following sample saturation, the extraction and injection manifolding, if necessary, are filled with fluid while the expansion tank is only partially filled with fluid. The pressure monitoring portion of the system is filled with water and the differential pressure transmitter is set to about 2 kPa by adjusting the height of water in the column above it. Pressure is then applied to the expansion tank by regulated nitrogen gas; pressure control is achieved by very slowly bleeding the gas off.

Once the cell is pressurized, the experiment begins and the computer-controlled data acquisition and control system is started. The general program flowchart is presented in Figure 3.13 and the subroutine flowchart is shown in Figure 3.14. The set temperatures for the upper and lower copper sealing plates as well as the operating pressure and temperature limits are keyed into the computer. With the experiment in operation the cooling

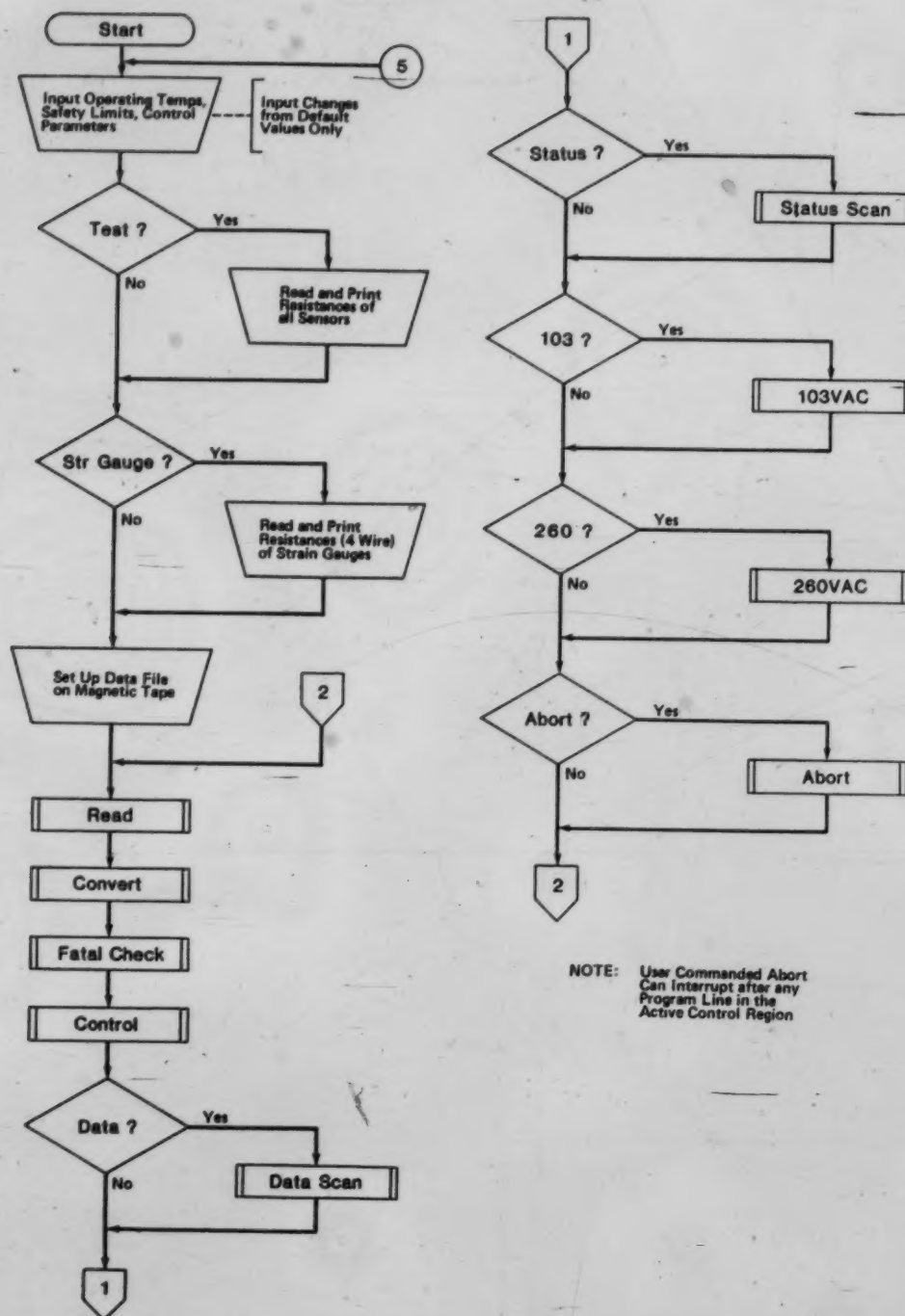


FIGURE 3.13 BASIC FLOW CHART OF DATA ACQUISITION AND CONTROL PROGRAM

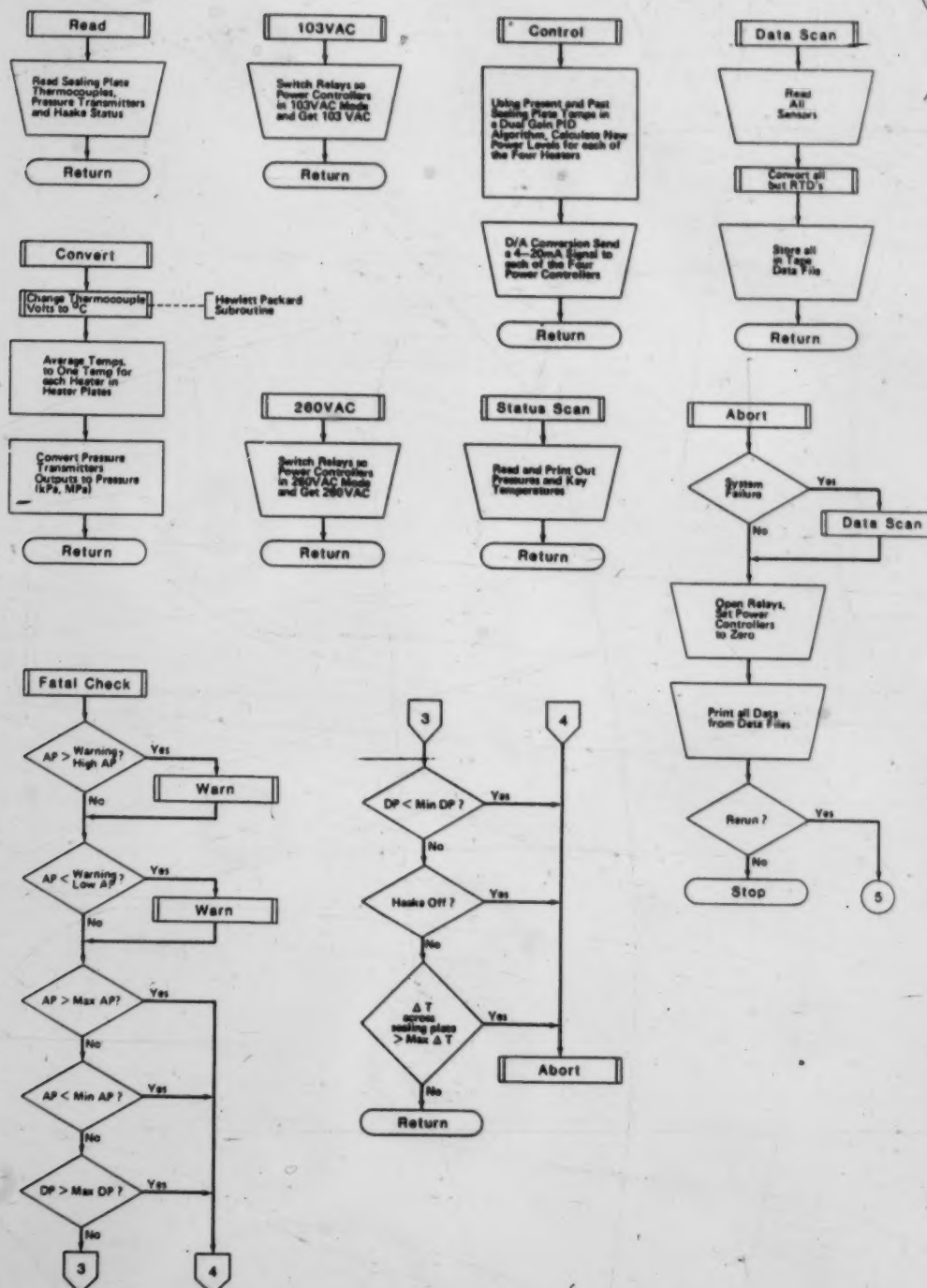


FIGURE 3.14 SUBROUTINE FLOW CHART OF DATA ACQUISITION AND CONTROL PROGRAM

oil circulator bath temperature and the water cooling flow rates are adjusted so that the heaters on the cold end of the guard and the cold heating/cooling head would act only as trimmers. For example, the Haake circulator temperature is usually set about 25°C below the cold end set point, and the guard heater temperature controllers are set so that the guard temperature is the same as or slightly above the temperature of the cell wall.

Once the coolers are set correctly, the experiment is checked occasionally with status requests, and when steady state conditions exist one to four data runs are made. Steady state conditions usually required about a day to appear. The cell is brought up to operating temperatures with 260 VAC on the main heaters, and 103 VAC is then used to reduce fluctuations at steady state.

Upon completion of an experiment the computer program is aborted and a new pair of upper and lower copper sealing plate temperatures are selected. The pore fluid pressure is then adjusted to maintain pore fluids in the liquid phase and a new experiment is begun. Upon completion of the entire experimental series the program is aborted and the cell is cooled down by completely opening the water cooler valves. The cell is then disassembled. The depths of the temperature probes are then measured by using a depth gauge mounted on a face plate. The differences between the depths are the sample thicknesses and the

thickness between each pair of temperature sensors is divided by the temperature difference and the resulting ratios are averaged and substituted into the calibration equation (4.2.1). Sampling for analysis of the cell test sample is then done if necessary. An entire experimental series of preparing the cell, packing, testing, sampling and clean up usually took approximately one month.

4. HEAT TRANSFER CELL TESTING AND PRELIMINARY RESULTS

4.1 Preliminary Testing and Modifications

Preliminary testing of the heat transfer cell comprised of room temperature pressure tests and temperature control system checks. The first hydrostatic pressure test was terminated at 4.2 MPa due to the development of pinhole leaks in the copper sealing plate as a result of the copper plate injection ports being drilled too close to the heater grooves. The leaking ports were silver-soldered shut and new ports were drilled. The second hydrostatic test was terminated at 8.4 MPa due to brittle fracture of the cell walls. The asbestos-cement pipe was rated at 4.9 MPa; thus the pipe pressure limit was set to 4.9 MPa, and the maximum temperature to 260°C, the corresponding water saturation temperature. Also, it should be noted that fracture did not occur at the cell wall probe ports, indicating that the probe port fittings reinforced the cell wall satisfactorily. The final hydrostatic test was cyclic, with pressures ranging from atmospheric to 4.9 MPa with a period of about one day and a total duration of two weeks. The cell survived this test without leaks.

The temperature control system was tested by first running the empty cell at elevated temperatures, and then by running the cell at elevated temperatures with the heat flux standards in place

and using a thin disc of Pyrex 7740 as the sample. The first test is used to check the main heaters, main cooler, and the basic utility of the control program. The second test checked the guard system and the temperature distribution throughout the cell.

Initially, the main heaters were simply fiberglass-sheathed nichrome wire glued into "V" grooves on the back of the copper sealing plates with a high temperature ceramic adhesive (Sauereisen Insa-lute Number 1). However, when the heaters were turned on, thermal expansion of the wire caused shorts and after three unsuccessful repairs, two new heating plates using tubular heaters were constructed. Also, during the many assemblies and disassemblies of the heating/cooling heads, the cooling plate began to leak around its gasket. The 0.8 mm thick compressed asbestos gaskets was successfully replaced by 3.2 mm thick compressed asbestos gaskets installed with Loctite 515 Gasket Eliminator.

The test runs with the thin Pyrex disc allowed tuning of the main heater PID control and partial tuning of the guard heater PID control. The runs used RTDs on the heat flux meters and E-type thermocouples on the thin disc. These runs also revealed problem areas that were corrected between runs or after the thin disc series was completed. For example, a thermocouple card and the temperature controllers for the guard and pore fluid heaters were

damaged and repaired, and the two multiplexer cards for RTD measurement had to be reconfigured to measure the 100 ohm RTDs.

Angular variations of temperature over the lower surface of the thin pyrex disc were studied. For a run with copper sealing plates temperatures of 100°C and 150°C the angular variations at a constant radius were a maximum of $\pm 0.75^\circ\text{C}$ versus the radial temperature variation of $\pm 1.6^\circ\text{C}$. The radial temperature variations in the upper copper sealing plate for the same run were $\pm 1.7^\circ\text{C}$. Angular temperature variations were less than half the radial temperature variations and therefore were not studied after this series of runs.

The copper sealing plate temperatures were very stable with time once steady state was achieved, and varied less than $\pm 0.1^\circ\text{C}$. However, there were significant temperature variations radially across the copper sealing plate. The radial temperature distributions from one of the runs is shown in Figure 4.1. The form of the distributions was consistent from run to run and the radial variation of upper copper sealing plate temperature from the set point for various runs can be approximated by:

$$T_{\text{ACT}} = T_{\text{SET}} \pm 0.01 \{ (T_{\text{SET}} - 33) + \Delta T_{\text{SET}} \} (^\circ\text{C}) \quad 4.1.1$$

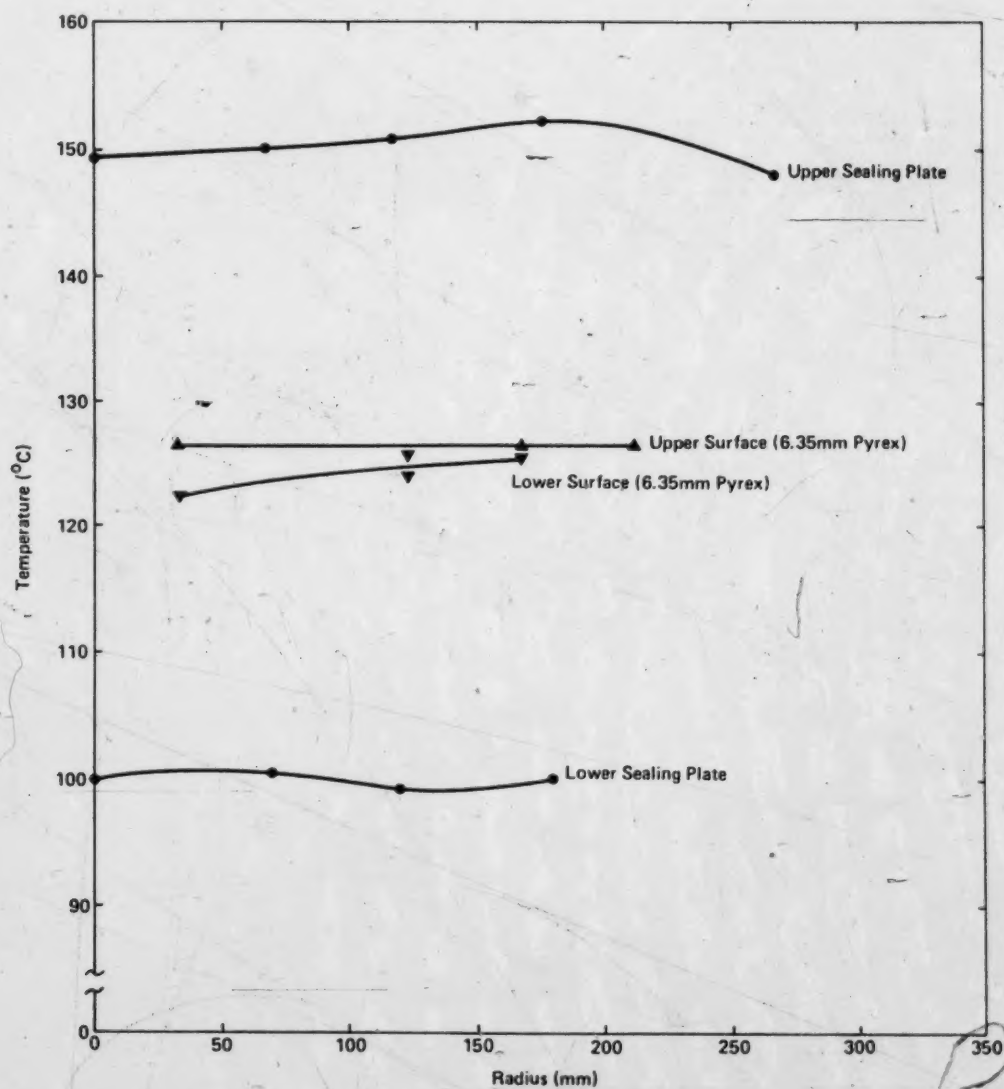


FIGURE 4.1 INITIAL RADIAL TEMPERATURE DISTRIBUTIONS

where

T_{ACT} = actual temperature of copper sealing plate ($^{\circ}C$)

T_{SET} = set temperature of copper sealing plate ($^{\circ}C$)

$\Delta T_{SET} = T_{hot \text{ sealing plate}} - T_{cold \text{ sealing plate}}$

Since the variations were consistent, their cause was also likely consistent and was probably due to inadequate guarding; thus the guarding system was improved. After the series was completed the guard system was revamped with greater heating capacity and greater thermal contact from the cold flange to the cold end of the guard. Also, a fragile multipoint thermopile initially used for radial temperature difference measurements was replaced with an amplified two point differential thermocouple in order to reduce breakage problems.

4.2 Calibration

Following the thin disc series, a series of cell calibration runs utilizing a 31.75 mm thick Pyrex disc was begun. In these tests the heat flux meters used RTDs while the calibration disc used had E-type 36 gauge thermocouples. This series had a threefold purpose: to calibrate the test cell for thermal conductivity measurements, to fine-tune the main heater PID control, and to improve the guard system.

After the initial test with the thick disc, a counter-current flow cooler was installed on the cold end of the guard. The cooler was initially supplied cooling oil from the main coolers. However, the next test showed the cooling oil circulator could not handle the additional load, and the guard cooler was connected to the water supply. The test also showed the need for more and larger guard heaters. The next test finalized control values for the guard temperature controllers. Figure 4.2 shows the effect of the guarding system changes on the radial temperature distribution of the upper copper plate. This is where the largest variation in temperature had been noted.

Main heater PID control optimization progressed throughout the test series and by the series end the steady state temperature variation with time was $\pm 0.01^{\circ}\text{C}$.

The calibration runs with the thick disc were conducted over an average temperature of 75°C to an average temperature of 242.5°C , where this average temperature is the average of the hot and cold copper plate temperatures. The difference between the hot and cold copper plate temperatures was either 45°C or 50°C for each test and the cold copper plate temperatures ranged from 50°C to 220°C while the hot copper plate temperatures ranged from 100°C to 265°C . Three calibration equations were developed from this run.

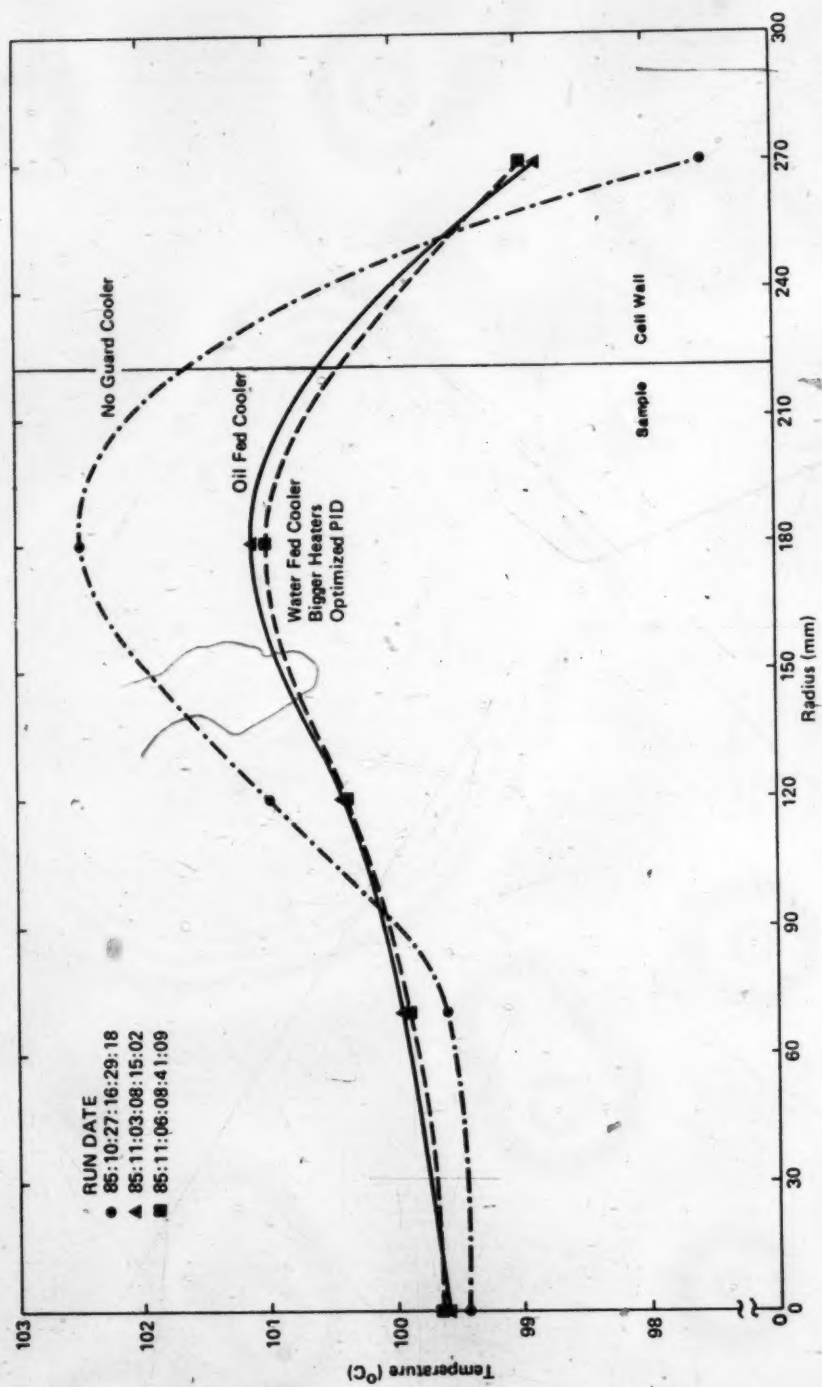


FIGURE 4.2 UPPER COPPER PLATE TEMPERATURE DISTRIBUTIONS

They are:

$$\lambda_{\text{SAM}} = \frac{C_{\text{UL}} t_{\text{SAM}}}{2\Delta T_{\text{SAM}} t_{\text{HFM}}} \{ \lambda_{\text{UHFM}} \Delta T_{\text{UHFM}} + \lambda_{\text{LHFM}} \Delta T_{\text{LHFM}} \} \quad 4.2.1$$

where:

$$C_{\text{UL}} = 1.03 - 8.33 \times 10^{-4} T_{\text{AV}} + 8.93 \times 10^{-6} T_{\text{AV}}^2,$$

$$\lambda_{\text{SAM}} = \frac{C_{\text{U}} t_{\text{SAM}}}{\Delta T_{\text{SAM}} t_{\text{HFM}}} \{ \lambda_{\text{UHFM}} \Delta T_{\text{UHFM}} \} \quad 4.2.2$$

where:

$$C_{\text{U}} = 1.16 - 9.41 \times 10^{-4} T_{\text{AV}} + 1.01 \times 10^{-5} T_{\text{AV}}^2,$$

$$\lambda_{\text{SAM}} = \frac{C_{\text{L}} t_{\text{SAM}}}{\Delta T_{\text{SAM}} t_{\text{HFM}}} \{ \lambda_{\text{LHFM}} \Delta T_{\text{LHFM}} \} \quad 4.2.3$$

where:

$$C_{\text{L}} = 0.927 - 7.50 \times 10^{-4} T_{\text{AV}} + 8.04 \times 10^{-6} T_{\text{AV}}^2$$

and where:

λ_{SAM} = sample thermal conductivity (W/mK)

λ_{UHFM} = upper heat flux meter thermal conductivity at its average temp (W/mK)

λ_{LHFM} = lower heat flux meter thermal conductivity at its average temp (W/mK)

ΔT_{SAM} = temperature drop through sample (°C)

ΔT_{UHFM} = temperature drop through upper heat flux meter (°C)

ΔT_{LHFM} = temperature drop through lower heat flux meter (°C)

t_{SAM} = sample thickness across which ΔT_{SAM} is measured (m)

- t_{HFM} = heat flux meter thickness between sensors (0.02975 m)
 C_{UL} = calibration factor when using both upper and lower heat flux meters
 C_U = calibration factor when using only upper heat flux meter
 C_L = calibration factor when using only lower heat flux meter
 T_{AV} = average of upper and lower copper plate temperatures ($^{\circ}C$)

4.3 Thermal Conductivity Tests

4.3.1 Ottawa Sand - Water Number 1

Following the calibration series, the first test of the complete heat transfer cell was begun using heat flux meters and probes that were fitted with 100 ohm RTDs. The sample for the test used 70-140 mesh Ottawa sand poured dry through the distributor into the cell. Water saturation was attempted by pulling a vacuum on the cell and injecting de-ionized and de-aired water into the cell. Post-run examination showed that very little water had entered the sandpack, probably due to water bypassing the sand pack and flowing up the cell walls.

The heat flux meters were 31.75 mm thick one-piece discs mounted on pads of silicone rubber. These pads were used in an effort to reduce cracking problems from uneven overburden pressure distribution of the sample onto the heat flux meters. The pads were made by gluing the heat flux meters to their respective

copper sealing plates with a thin layer of silicone rubber caulking. Under the conditions of limited air contact the rubber did not fully cure and when the pads were subjected to high temperatures, reversion occurred and the rubber broke down. The pad breakdown resulted in severe heat flux meter cracking and most of the heat flux meter RTDs failed.

The gaskets used were the same as those used in the preliminary pressure tests: Flexitallic 18" 150-pound spiral wound asbestos-stainless steel. However, deformation of the copper sealing surfaces made very large seating stresses of approximately 165 MPa necessary for a seal, and the cell wall cracked after 5 hours at 220°C and 2.6 MPa.

Thermal conductivities calculated from the test series were very rough because of the failure of the RTDs in the heat flux meters. The average thermal conductivity calculated was 0.7 W/mK. Cervenán, Vermuelen and Chute (1981) found a thermal conductivity of 0.8 W/mK for a sand pack containing 1%, by mass, of water.

The radial temperature distributions within the upper copper sealing plate were still not adequately uniform, with a variation of $\pm 1.2^\circ\text{C}$ within the sample area.

To attempt to solve the various problems found in this test, several changes were made. To reduce heat flux meter cracking

the heat flux meters were cut into 6 equal wedges and the silicone rubber pad was made with Dow Corning 3120 RTV, a deep section curing, silicone rubber rated for 300°C. To correct for high RTD failure rates, RTDs in both the heat flux meters and the probes were replaced with E-type thermocouples. Improvements in water saturation were sought by shortening the injection tubing and removing vapour-trapping vertical bends.

New 6.35 mm thick custom Flexitallic gaskets were installed. The gaskets had no carbon steel supporting ring and were made larger in outside diameter so that the copper sealing plate would supply blow-out protection. The thicker gasket was used in an attempt to reduce necessary gasket sealing stress to a level where the asbestos-cement walls of the cell would not fail from excessive compression.

The problem of excessive radial temperature variations in the upper sealing plate was attacked by two methods. The first method connected the injection ports in pairs, thereby halving the number of injection tubes leaving the copper sealing plates and the cell. The second method ran the guard at higher offset temperatures than the 0°C offset from the cell walls than the 0°C previously used.

4.3.2 Ottawa Sand - Water Number 2

This run was essentially a repeat of the first, with changes as already noted.

Vacuum saturation was again tried, but calculated thermal conductivities suggest it was unsuccessful. Sampling of the sand pack was not possible as a simulated oil sand run was conducted using the same sand pack without cell disassembly.

Cracking of the heat flux meters was considerably reduced and only 4 of 28 temperature points of importance were lost during the run.

The new gaskets reduced the gasket seating stresses to approximately 106 MPa which was still too high; therefore the cell assembly was not run at the highest pressures and temperatures. The maximum temperature used was 200°C, the maximum pore pressure was 1.7 MPa, and the overburden pressure was 5.4 MPa.

Thermal conductivities were calculated from the test series and are shown in Table 4.1. The estimated accuracy of the effective thermal conductivity measurement was originally estimated as $\pm 5.6\%$, but temperature variations within the cell increased the estimated error to $\pm 7.2\%$. Error calculations are illustrated in

Appendix E. A water content of 0.8% mass percent was calculated from a mass balance. Cervenán, Vermuelen and Chute (1981) found a thermal conductivity of 0.8 W/mK for a sand with 1.0% water, by mass, at 25°C and atmospheric pressure.

Table 4.1 Ottawa Sand-Water Number 2 Thermal Conductivity

Run	T _{UPPER} (°C)	T _{LOWER} (°C)	λ_{SAMPLE} (W/mK)
2	100	55	2.80*
6	125	75	0.58
7	150	100	0.72
8	175	125	0.68
9	200	150	0.76

*not at steady state

The variation between the thermal conductivities of the last four runs was ± 0.1 W/mK which is $\pm 14.6\%$. These variations in effective thermal conductivity were probably due to the migration of the small amount of water present. This moisture migration could cause small variations because steady state would not have been truly reached. The generally increasing trend of the measured thermal conductivities with temperature is supported by Somerton, Keese and Chu (1974). They found that porous materials with thermal conductivities of less than 1.42 W/mK at 52°C had

their thermal conductivity increase with temperature.

4.3.3 Simulated Oil Sand

The simulated oil sands series of runs was conducted by first flooding the previously used sand pack with Cold Lake bitumen by flowing two pore volumes of bitumen through the sand pack. Bitumen injection and extraction was done from different ports in order to thoroughly sweep the cell. Bitumen at 90°C was pumped in from a piston-type accumulator which was in turn driven by a liquid chromatography pump. The mass of extracted fluid was measured in order to do a mass balance.

The radial temperature variations in the upper copper plate for one of the calibration runs and one of the runs in this series is shown in Figure 4.3. The maximum temperature variation from set point within the sample area was reduced from 2.45°C to 0.33°C.

Only three runs were done in this series and the resulting thermal conductivities are shown in Table 4.2. The maximum pore pressure was 1.6 MPa and the overburden pressure was 5.4 MPa.

The sand pack was sampled after the series was complete. The results are shown in Table 4.3.

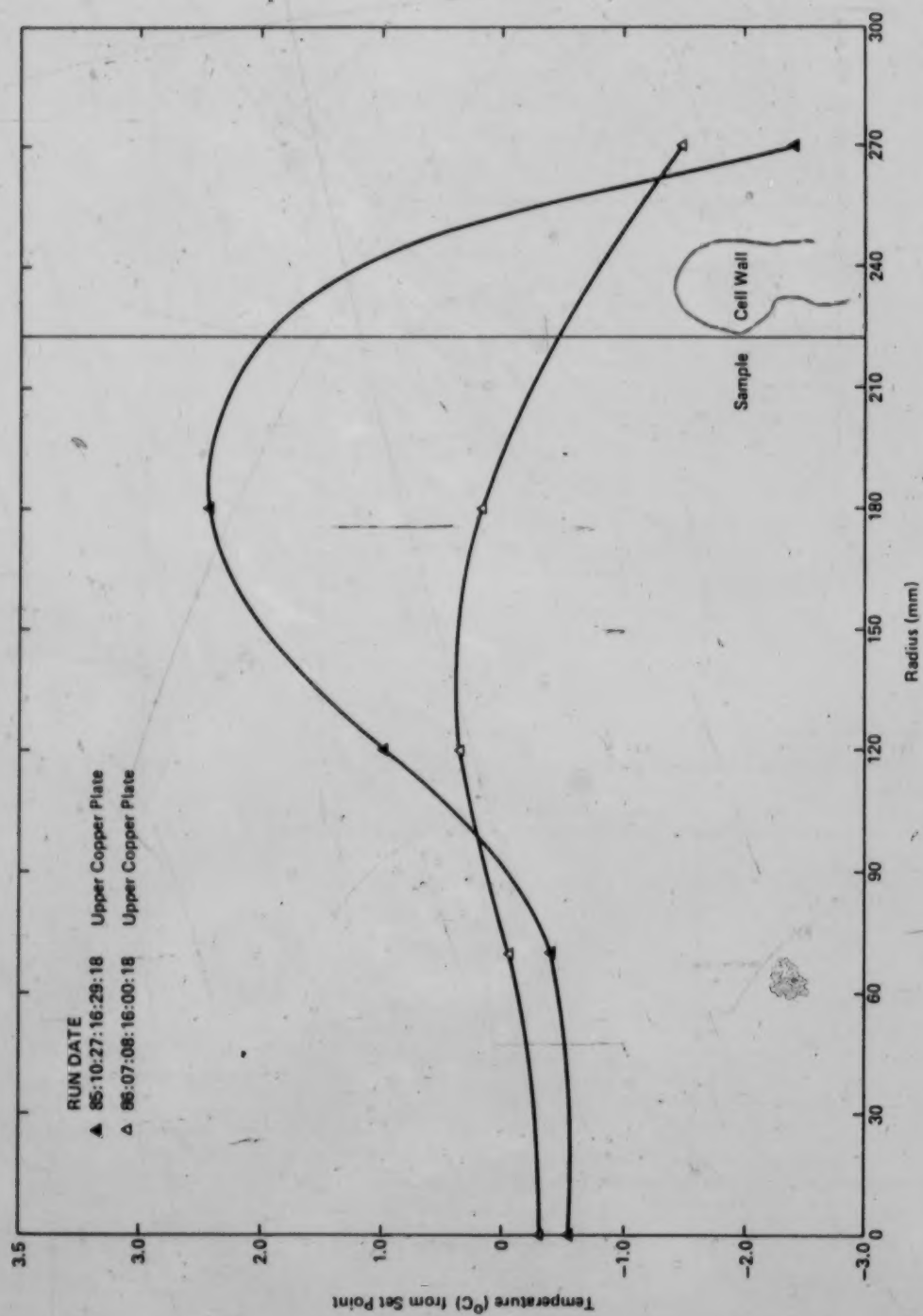


FIGURE 4.3 RADIAL TEMPERATURE VARIATIONS FROM SET POINT

Table 4.2: Simulated Oil Sand Thermal Conductivity

Run	T _{UPPER}	T _{LOWER}	λ_{SAM} (W/mK)
6	125	75	2.39*
7	175	125	1.29
8	175	125	1.34

* not at a steady state

Table 4.3: Simulated Oil Sands Saturations

Radius (mm)	Mass % Bitumen	Mass % Water	Mass % Sand	Comments
0	10.1	0.3	89.6	Clean sand lens 6 mm deep x 30 mm diameter centrally located axially and radially
140	13.4	0.4	86.3	
165	12.7	0.5	86.6	
184	12.3	0.6	87.1	
203	12.7	1.0	86.3	Top of pack
203	13.3	0.8	85.9	Bottom of pack
75-200	13.6	0.1	86.1	Bulk sample

The average mass composition of the simulated oil sand pack was 12.6% bitumen, 0.5% water, and 86.8% sand. The bitumen occupied 79.0% and the water occupied 3.2% of the sand pack pore space. Cervenán, Vermuelen and Chute (1981) found a thermal conductivity of 1.31 W/mK for a reconstituted oil sand sample of 13.9% bitumen, 0.8% water, and 85.3% sand at 25°C and atmospheric pressure. This agrees very closely with the results of runs 7 and 8 (1.29 and 1.34 W/mK). Scott and Seto (1985) measured a thermal conductivity of 1.75 W/mK for an undisturbed oil sand sample containing 12.3% bitumen, 4.5% water, and 83.2% sand at 150°C and a pore pressure of 2 MPa. The reasons Scott and Seto measured a higher thermal conductivity are most likely the better packing of the undisturbed oil sand and the complete saturation of the pore spaces with fluids.

The large value of thermal conductivity for run 6 was most likely due to the data run not being taken at steady state and the absorption of heat into the sample giving the appearance of high thermal conductivity. Steady state was most probably not achieved because of two factors; the slow rate of heating available to the sample once the sealing plates reached their set temperatures, and the sample was being heated from room temperature to about 100°C, unlike the other experiments where its temperature was only being increased 25°C.

5. CONCLUSIONS AND RECOMMENDATIONS

The overall objective was to design, build, and test a heat transfer cell. The cell was to be capable of measuring the effective thermal conductivity of oil sands material under in situ like conditions and to have the capability of being used to study macroscale natural convection in an oil sands-like system under similar conditions. This study was limited to the design and construction of the cell, and only testing in the thermal conductivity mode was done.

It is concluded that:

1. The developed cell has a temperature range of 25°C to 260°C, a pore pressure range of 0.1 MPa to 2.0 MPa and an overburden pressure range of 1 MPa to 6 MPa.
2. Preliminary measurements of the effective thermal conductivity of Ottawa sand, water, and bitumen mixtures over a range of temperatures, pore pressures, and overburden pressures were found to be in close agreement with published values for similar mixtures and in situ like conditions.
3. The estimated accuracy of the effective thermal conductivity measurement is $\pm 7\%$.

4. The useable operating ranges of temperature and pressure were limited due to deformation of the sealing surfaces of the copper sealing plate.

It is recommended that modifications are still needed for the heat transfer cell to develop its full potential for measuring both thermal conductivities of large oil sand samples at temperatures up to 260°C and pressures of up to 4.7 MPa, and to detect natural convection in oil sand-like systems. A proposed solution to the gasketing problem is to cover the gasket surfaces of the cell walls with stainless steel and to replace the copper sealing plate with a composite sealing plate that has stainless steel sealing surfaces and a thick copper insert. The thick copper insert would smooth the horizontal temperature distribution, just as the copper sealing plate did, while the stainless steel sealing surfaces would reduce distortion and therefore gasket sealing stress.

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APPENDIX A: NATURAL CONVECTION CALCULATIONS

The dimensions of the heat transfer cell must be such that natural convection can occur in it, under in situ conditions and with a reasonable temperature difference across the sand pack. The thickness of the sample layer was calculated so that the sand pack permeability necessary for convection was minimized. The dimensionless variable best characterizing natural convection in a porous medium is the Rayleigh number, as modified by Katto and Masuoka (1967):

$$Ra = \frac{g\beta \Delta T k\delta}{\alpha_m \nu} \quad A.1$$

where, for a water saturated 36% porosity Ottawa:

g = local gravity acceleration constant = 9.81 m/s^2

β = coeff. of volume expansion of water = $7.2 \times 10^{-4} \text{ 1/K}$

ΔT = temp. difference across the layer = 230°C (max.)

k = permeability of the water-saturated porous medium
 $= k \times 10^{-12} \text{ m}^2$ for a sand of "k" darcy permeability

δ = thickness of the layer (m)

ν = kin. viscosity of water = $8.068 \times 10^{-7} \text{ m}^2/\text{s}$ @ 30°C

α_m = thermal diffusivity of the medium (m^2/s)

where

$$\alpha_m = \frac{\lambda_m}{(\rho C_p)_f}$$

where:

λ_m = thermal conductivity of the saturated porous medium
(W/mK)

= 2.05 W/mK for water sat. 36% porosity Ottawa sand

ρ_f = density of water = 995.67 kg/m³ @ 30°C

C_{p_f} = specific heat-capacity of water = 4176 J/kg °C
@ 30°C

Substituting values gives:

$$Ra = 4.033 \delta k$$

A.2

At a critical value of Ra (Ra_c), buoyant forces will override viscous forces and convection will begin.

If viscosity is constant and the layer of the porous medium is horizontally infinite, Lapwood (1948) showed:

$$Ra_c = 4\pi^2$$

However, for a finite volume of porous medium saturated with water, which has a temperature-dependent viscosity, certain corrections must be made. Kassoy and Zebib (1975) analyzed the effects of variable water viscosity on the onset of convection in porous media. They presented the following method for estimating Ra_c when there are viscosity effects:

$$\tau = \frac{T_1 - T_o}{T_o}$$

A.3

where:

τ = dimensionless ratio characterizing variable viscosity effect

T_o = upper surface temp.,

T_o = lower surface temp.

Ra_c^v - variable viscosity effects included.

The following table was calculated by Kassoy and Zebib (1975) for water in porous medium.

Table A.1: Viscosity Effects on Onset of Natural Convection

τ	Ra_c^v
0	$4\pi^2$
.34	17.25
.50	13.35
.67	10.76
.84	8.86

For the cell under consideration

$$T_o(\text{min}) = 303\text{K}$$

$$T_i(\text{max}) = 533\text{K}$$

$$\tau(\text{max}) = 0.76$$

$$\therefore Ra_c^v(\text{min}) = 9.75$$

However, Ra_c is not 9.75 unless the layer of porous medium is of infinite extent. A finite medium is corrected for by using an aspect ratio correlation developed by Zebib (1977), which is:

$$Ra_c^{AR} = \left(\frac{\gamma^2 + \pi^2}{\gamma} \right) \quad A.4$$

Zebib claimed that viscosity effects and aspect ratio effects are independent, so the effects will simply be superimposed.

$$Ra_c^{VAR} = Ra_c^v \cdot \left[\frac{Ra_c^{AR}}{4\pi^2} \right] \quad A.5$$

where:

$Ra_c^{VAR} = Ra_c$ as calculated with both aspect ratio and variable viscosity effects

$Ra_c^{AR} = Ra_c$ as calculated with aspect ratio effects but not viscosity effects

$4\pi^2 = Ra_c$ with no aspect ratio effects or viscosity effects.

substituting values gives:

$$Ra_c^{VAR} = \frac{9.75}{39.48} Ra_c^{AR}$$

$$\therefore Ra_c^{VAR} = .247 Ra_c^{AR}$$

Zebib found that if the aspect ratio is less than 0.582 i.e.,

$$\frac{\delta}{r} < 0.582$$

then:

$$\gamma = \frac{1.841}{r} \delta$$

where r = radius of porous media = 0.23 m

$$\therefore \gamma = 8 \delta$$

A.6

$$\text{and } Ra_c^{AR} = \left[\frac{64\delta^2 + \pi^2}{8\delta} \right]^2$$

$$\text{thus } Ra_c^{VAR} = .247 \left[\frac{64\delta^2 + \pi^2}{8\delta} \right]^2$$

For the cell design conditions (eqn. A.2)

$$Ra = 4.03 \delta k = Ra_c^{VAR}$$

Substituting gives:

$$4.03 \delta k = .247 \left[\frac{64\delta^2 + \pi^2}{8\delta} \right]^2$$

Therefore:

$$k = \frac{.0612}{\delta} \left[\frac{64\delta^2 + \pi^2}{8\delta} \right]^2$$

$$k = \frac{.0612}{64} (\delta^{-3}) (64\delta^2 + \pi^2)^2$$

$$\frac{dk}{d\delta} = \frac{0.0612}{64} (-3\delta^{-4} (64\delta^2 + \pi^2)^2 + 2\delta^{-3} (64\delta^2 + \pi^2)128\gamma)$$

for minimum permeability of sand:

$$\frac{dk}{d\delta} = 0 = \frac{(64\delta^2 + \pi^2)}{\delta^2} \left[\frac{-3(64\delta^2 + \pi^2)}{\delta} + 256 \right] \quad \text{A.7}$$

$$\frac{256}{3} \delta^2 = 64\delta^2 + \pi^2$$

therefore:

$$\delta = .68 \text{ m.}$$

$$k = 4.74 \text{ darcy.}$$

$$r = 0.23 \text{ m.}$$

$$Ra_c^{\text{VAR}} = 13.0$$

APPENDIX B: HEAT TRANSFER CALCULATIONS

Following is a simple analysis of the heat flows throughout the cell.

Nomenclature

- λ_{OS} = oil sands thermal conductivity = 2 W/mK (maximum)
- λ_{ST} = steel thermal conductivity = 50 W/mK
- λ_{HFM} = pyrex 7740 thermal conductivity = 2.2 W/mK
- λ_{AC} = asbestos-cement thermal conductivity = 0.6 W/mK (est)
- λ_{INS} = insulation thermal conductivity = 0.08 W/mK (worst case)
- D_0 = diameter of A/C pad = 635 mm
- D_1 = internal diameter of cell = 60 mm
- D_2 = outer diameter of cell = 570 mm
- D_3 = internal diameter of guard = 746 mm
- D_4 = diameter of flange = 944 mm
- t_{INS} = insulation thickness
- t_{HFM} = heat flux meter thickness = 31.8 mm
- t_{OS} = oil sands thickness = 63.5 mm or 680 mm (conductivity mode or convection mode)
- t_G = guard thickness = 7.6 mm
- t_{AC} = asbestos-cement pad thickness = 25.4 mm
- t_H = heating cooling head thickness = 57.2 mm
- T_m = heating head temperature
- T_C = cooling head temperature

- T_{FH} = hot flange temperature
 T_{FC} = cold flange temperature
 T_G = guard temperature
 T_{AC} = asbestos-cement pipe temperature
 T_A = ambient air temperature
 Q_1 = heat flux (vertical) hot head to cold head through heat flux meter and sample
 Q_2 = heat flux (vertical) hot head to cold head through asbestos-cement pipe
 Q_3 = heat flux (vertical) head to flange through asbestos-cement pad
 Q_4 = heat flux (radial) asbestos-cement pipe to guard
 Q_5 = heat flux (radial) guard to ambient air
 Q_6 = heat flux (vertical) through guard
 Q_7 = heat flux (vertical) flange to ambient air

Note: Conduction heat transfer through composite material. All heat flows were assumed to be either vertical (axial) or radial, and independent of one another. Outer surfaces were assumed to be at ambient air temperatures.

$$Q_1 = \frac{(T_H - T_C) (\pi/4) D_1^2}{2 \left(\frac{t_{HFM}}{\lambda_{HFM}} \right) + \frac{t_{OS}}{\lambda_{OS}}}$$

B.1

$$Q_2 = \frac{\lambda_{AC} (T_H - T_C) (\pi/4) (D_2^2 - D_1^2)}{2 t_{HFM} + t_{OS}} \quad B.2$$

$$Q_3 = \frac{\lambda_{AC} (T_H - T_{FH}) (\pi/4) D_o^2}{t_{AC}} \quad B.3a$$

or

$$\frac{\lambda_{AC} (T_{FC} - T_C) (\pi/4) D_o^2}{t_{AC}} \quad B.3b$$

$$Q_4 = \frac{2\pi \lambda_{INS} (2t_{HFM} + t_{OS} + 2t_H + 2t_{AC})(T_G - T_{AC})}{\ln\left(\frac{D_3}{D_2}\right)} \quad B.4$$

$$Q_5 = \frac{2\pi \lambda_{INS} (2t_{HFM} + t_{OS} + 2t_H + 2t_{AC})(T_G - T_A)}{\ln\left(\frac{D_3 + 2t_{INS}}{D_3}\right)} \quad B.5$$

$$Q_6 = \frac{\lambda_{ST} \pi D_3 t_G (T_H - T_C)}{2 t_{HFM} + t_{OS}} \quad B.6$$

$$Q_7 = \frac{\lambda_{INS} (\pi/4) D_4^2 (T_{FH} - T_A)}{t_{INS}} \quad B.7$$

or

$$Q_7 = \frac{\lambda_{INS} (\pi/4) D_4^2 (T_{FC} - T_A)}{t_{INS}} \quad B.7b$$

The heat flows were found for two cases; a typical conductivity mode and a typical convection mode. The typical conductivity

mode has a 63.5 mm thick sample and a maximum temperature difference through sample and heat flux meters of 60°C. The typical convection mode has a 680 mm thick sample and a maximum temperature difference of 230°C.

Q_1

Conductivity mode: $Q_1 = 117.7 \text{ W}$

Convection mode: $Q_1 = 171.4 \text{ W}$

Q_2

Conductivity mode: $Q_2 = 26.5 \text{ W}$

Convection mode: $Q_2 = 17.3 \text{ W}$

Q_3

$Q_3 = Q_7 + \text{a portion of } Q_5 \text{ and } Q_6$

Q_4

max $\Delta T = 0.1^\circ\text{C}$ at top and bottom

6°C at centre

ΔT average = 3°C (approx.)

Conductivity mode: $Q_4 = 1.7 \text{ W}$

Convection mode: $Q_4 = 5.3 \text{ W}$

Q_5

$(T_G - T_A)_{\max} = 210^\circ\text{C}$ or 125°C

$2 t_{\text{INS}} = D_4 + 2 (.1524) - D_3 = 500 \text{ mm}$

Conductivity mode: $Q_5 = 60.2 \text{ W}$

Convection mode : $Q_5 = 111.5 \text{ W}$

Q_6

Conductivity mode: $Q_6 = 420.7 \text{ W}$

Convection mode: $Q_6 = 275.5 \text{ W}$

Q_7

$t_{INS} = 300 \text{ mm}$

$\Delta T = 240^\circ\text{C}$ $Q_7 \text{ max} = 44.4 \text{ W}$

$\Delta T = 10^\circ\text{C}$ $Q_7 \text{ min} = 1.9 \text{ W}$

This one-dimensional analysis is far from exact as the effect of the various coolers was ignored and the actual heat flows are more complex. However, it is sufficient to approximate the magnitudes of the heat fluxes. Once the approximate heat fluxes are known, heaters and coolers of appropriate sizes can be designed.

The main heating plates of the cell must have a high power capacity so as to rapidly warm the cell and avoid excessive segregation of pore fluids. However, they must also be able to control temperatures very accurately and stably at the low heat flux rates associated with the steady state. This is done by operating the heating plates on two different voltages, 260 VAC or 103 VAC. These values were chosen as the maximum voltage

difference that the power controllers could handle, with modifications.

At steady state conditions the hot end heating plate must supply Q_1 , Q_2 , Q_3 , and Q_4 ; the total of Q_1 , Q_2 , and Q_4 is a maximum of 200 watts. Q_3 is less than the sum of Q_5 , Q_6 and Q_7 , which is 525 watts and is thus used for Q_3 's upper bound. Therefore, steady state power demand on the hot end heating plate is less than 725 watts. However, a 725 watt heating plate would be on almost 100% of the time at high temperatures, and to reduce the duty cycle and also have an operating safety factor, a 1200 watt 103 VAC heating plate was selected. This plate would supply 7500 watts at 260 VAC. Using a simple lump heat capacity analysis, two of these heater plates would, at 260 VAC, heat the 1.4 tonne conductivity mode cell to maximum operating conditions in 2.6 hours and would heat the 1.7 tonne convection mode cell to operating conditions in 2.1 hours. Three tubular heaters were placed into each heating plate, with the inner one being 200 mm long and producing 3500 watts at 260 VAC, and the outer two, which were parallel connected, being 1600 mm long (2350 W) and 1200 mm long (1750 W). The heaters were bent into radiuses such that an equal power density is available across the heating plate. The outer heaters were bent to 270 mm and 220 mm radiuses and the inner heater was wound to radiuses of 55 mm, 110 mm and 170 mm.

The heaters for the guard are heat tapes 2400 mm in length, which is a single circumference of the guard. The hot end guard heater must be capable of supplying Q_6 and one half of Q_4 and Q_5 , which is 500 watts. The cold end guard heater must supply one half of Q_4 and Q_5 , which is 60 watts. However, the guard system deals with outside effects on the heat transfer in the cell and needs substantially additional capability. To meet this requirement the hot end guard heater is made of two heat tapes of 1250 watts total power and the cold end guard heater consists of two heat tapes of 400 watts total power. There is too much thermal resistance between the cold end of the guard and the cooling plate, so a guard cooler was installed to keep the cold end at the desired temperature. Water is run through two 6.35 mm diameter copper tubes in a parallel, counter-current arrangement. The tubes were embedded in heat transfer cement on the cold end of the guard. At the maximum flow rate possible of 1.5 litres per second, the total guard heater power would raise the water temperature 0.3°C from its normal 15°C.

The cooling plate must absorb Q_1 and Q_2 , which is 200 watts. The cooling head uses three parallel counter-current paths for the cooling oil (Dow Corning 210M). To avoid the use of valves on the paths they were designed with the same pressure drop at design flow rates. Valves were not used as the available pressure head is very low. The total pressure drop and total flow rate were calculated from the pump characteristic of the

Haake circulator pump. The paths were 22.2 mm wide, with the innermost and outermost being 2.77 mm deep and the middle path being 3.30 mm deep. The innermost path is 2930 mm long, the middle path is 3860 mm long, and the outermost path is 3370 mm long.

The energy carried by the fluid is:

$$Q_1 = C_p \rho V W D \Delta T \quad (W)$$

B.8

where:

$$C_p = \text{oil heat capacity @ } 25^\circ\text{C} = 1.515 \times 10^3 \text{ J/kg } ^\circ\text{C}$$

$$\rho = \text{oil density @ } 25^\circ\text{C} = 968 \text{ kg/m}^3$$

$$V = \text{velocity (average) (m/s)}$$

$$W = \text{path width} = 22.2 \times 10^{-3} \text{ m}$$

$$D = \text{path depth (m)}$$

$$\Delta T = \text{change in bulk fluid temperature } (^\circ\text{C})$$

substituting gives:

$$Q_1 = 3.256 \times 10^4 V D \Delta T \quad (W)$$

The energy input to the fluid is:

$$Q_2 = h (T_w - T_b) (2 (W + D)) L \quad (W)$$

B.9

where:

h = convection heat transfer coefficient ($\text{W/m}^2\text{°C}$)

T_w = wall temperature (°C)

T_b = bulk fluid temperature (°C)

L = path length (m)

From the Handbook of Heat Transfer (Rohsenow, Hartnett, 1973) for a rectangular channel:

$$h = \text{Nu} \lambda \left(\frac{D + W}{2DW} \right) \quad \text{B.10}$$

where:

$$\text{Nu} = 5.60 \text{ for } W/D = 8$$

$$4.80 \text{ for } W/D = 6.7$$

substituting gives:

$$Q_2 = 0.16 \text{ Nu } (T_w - T_b) (D + W)^2 L / WD \quad (\text{W})$$

The path pressure drop is:

$$\Delta P_1 = 2 f \rho V^2 L \left(\frac{W + D}{2WD} \right) \quad (\text{Pa}) \quad \text{B.11}$$

where:

f = friction factor

for the laminar flow regime the cooler operates in, the friction factor is:

$$f = 20.5/Re$$

where

$$Re = \frac{\rho V \sqrt{4WD}}{\mu \sqrt{2(W+D)}}$$

B.12

$$\mu = \text{oil viscosity @ } 25^{\circ}\text{C} = 0.1 \text{ kg/ms}$$

substituting gives:

$$\Delta P_1 = 2.08 \times 10^3 VL \frac{(W+D)^2}{D^2} \text{ (Pa)}$$

Inner Cooler:

covers from the centre to a radius of 160.1 mm

Q_i = heat flow in inner cooler (W)

$$Q_i = 200 (160.1/290)^2 = 61.0 \text{ W}$$

$$D = 2.77 \times 10^{-3} \text{ m}$$

$$L = 2.93 \text{ m}$$

noting:

$$Q_i = Q_{1i} = Q_{2i} = 61.0 \text{ W}$$

and substituting gives:

$$V_i \Delta T_i = 0.676 \text{ m}^3 \text{C/s}$$

$$(T_w - T_b)_i = 2.29 \text{ }^\circ\text{C}$$

$$\Delta P_{1i} = 4.95 \times 10^5 \text{ V}_i \text{ Pa}$$

Middle Cooler:

covers from a radius of 160.1 mm to a radius of 242.9 mm

Q_m = heat flow into middle cooler (W)

$$Q_m = 200 (242.9^2 - 160.1^2) / 290^2 = \underline{79.4 \text{ W}}$$

$$D = 3.30 \times 10^{-3} \text{ m}$$

$$L = 3.86 \text{ m}$$

noting:

$$Q_m = Q_{1m} = Q_{2m} = 79.4 \text{ W}$$

and substituting gives:

$$V_m \Delta T_m = 0.739 \text{ m}^\circ\text{C/s}$$

$$(T_w - T_b)_m = 3.02 \text{ }^\circ\text{C}$$

$$\Delta P_{1m} = 4.79 \times 10^5 \text{ V}_m \text{ Pa}$$

Outer Cooler:

covers from a radius of 242.9 mm to a radius of 290 mm

$$Q_o = Q_{1o} = Q_{2o} = 59.7 \text{ W}$$

and substituting gives:

$$V_o \Delta T_o = 0.662 \text{ m}^\circ\text{C/s}$$

$$(T_w - T_b)_o = 1.95 \text{ }^\circ\text{C}$$

$$\Delta P_{1o} = 5.70 \times 10^5 \text{ V}_o \text{ Pa}$$

Pressure drops must match, therefore:

$$\Delta P_{1i} = \Delta P_{1m} = \Delta P_{1o} = \Delta P \text{ (Pa)}$$

therefore:

$$V_i = 1.151 V_o \quad (\text{m/s})$$

$$V_m = 1.190 V_o \quad (\text{m/s})$$

substituting these gives:

$$\Delta T_i = 0.887 \Delta T_o \quad (^\circ\text{C})$$

$$\Delta T_m = 0.938 \Delta T_o \quad (^\circ\text{C})$$

The total flow rate is:

$$F = W(D_i V_i + D_m V_m + D_o V_o) \quad (\text{m}^3/\text{s})$$

B.12

$$F = 2.19 \times 10^4 V_o \quad (\text{m}^3/\text{s})$$

The pressure drop is:

$$\Delta P = 5.70 \times 10^5 V_o \quad (\text{Pa})$$

The pump characteristic for the Haake circulator must be compensated, as Dow Corning 210H silicone oil is being used instead of water. Daugherty and Franzini (1977) gave a figure from which the effects of the 100 centistoke fluid could be estimated and at the same flow rate the head produced is reduced to about 75% of the value for water. The pump characteristic for water in the Haake circulator can be approximated as:

$$\Delta P = 8.5 \times 10^4 - 3.48 \times 10^8 F \quad (\text{Pa}) \quad \text{B.14}$$

when corrected for oil it is:

$$\Delta P_{\text{OIL}} = 6.4 \times 10^4 - 2.6 \times 10^8 F \quad (\text{Pa}) \quad \text{B.15}$$

The circulator must supply head equal to the system pressure drop, therefore:

$$5.70 \times 10^5 V_o = 6.4 \times 10^4 - 2.6 \times 10^8 (2.19 \times 10^{-4} V_o)$$

therefore:

$$V_o = .102 \text{ m/s}$$

and therefore:

$$\Delta P = 58 \text{ kPa}$$

$$\Delta T_i = 5.8^\circ\text{C}$$

$$\Delta T_m = 6.1^\circ\text{C}$$

$$\Delta T_o = 6.5^\circ\text{C}$$

All of these calculations have been done for an oil temperature of 25°C. At higher oil temperatures the oil viscosity drops rapidly and cooling capacity is increased.

APPENDIX C: THERMAL EXPANSION CALCULATIONS

The areas of the most significant possible thermal expansion effects were the heating/cooling head sealing, cell sealing and sample expansion.

The heating/cooling head has a copper sealing plate and stainless steel heating and cooling plates. The very high thermal conductivity copper is used to provide a near isothermal surface, while the stainless steel is used for strength and for corrosion resistance to the cooling fluids used in the cooling head. At room temperature stainless steel has a linear coefficient of thermal expansion of 1.73×10^{-5} 1/K while the copper has a coefficient of 1.66×10^{-5} 1/K. At the maximum radius of 320 mm and a maximum temperature change of 235°C, the room temperature coefficients give a difference in expansion of 5.4×10^{-2} mm, which should not tear sealing gaskets.

Sealing of the cell could fail at elevated temperatures if the carbon steel studs compressing the cell extended more than the asbestos-cement pipe enough to unload the gaskets. The coefficient of linear thermal expansion for carbon steel is 1.2×10^{-5} 1/K while that for asbestos-cement pipe was approximated by concrete's value of 1.1×10^{-5} 1/K. The flanges are up to 907.8 mm apart and they compress 50.8 mm of asbestos-cement pad, 75.4 mm of stainless steel, 38.1 mm of copper, and up to 743.5 mm of

asbestos-cement pipe. The total difference in expansion with a 235°C temperature change was 5.2×10^{-2} mm, which was well within the flexibility of the seal of the two Flexitallic gaskets which are each compressed about 0.8 mm to seal.

J.D. Scott and K.M. Kosar (1982) showed bitumen expansion to be less than that of water for temperature changes from room temperature to over 135°C. The expansion of the solids in the sample is matched by the cell expansion but the pore fluids are allowed to flow into the pore fluid-control system. The density of water decreases 21.6% from 25°C to 260°C, increasing the volume of a constant mass of water by 27.5%. The design pore volume of the pack is about 36% of the pack volume and therefore the amount of water that would be forced out of the cell is about 10% of the original pack volume. The volume of the conductivity-mode pack volume is at most $1.46 \times 10^{-2} \text{ m}^3$ and fluids produced would be less than 1.5 litres. The volume of the convection-mode pack is $1.12 \times 10^{-1} \text{ m}^3$ and fluids produced as a result of expansion would be at most 11.2 litres. Thus expansion tanks were sized to contain 1.5 times the maximum estimated fluid production.

APPENDIX D: MECHANICAL CALCULATIONS

The major mechanical considerations were pressure containment and sealing, particularly of the cell and the heating/cooling head.

The cell is sealed by two 24" 600-lb blind flanges pressing copper sealing plates containing spiral wound steel and asbestos gaskets against the ends of the asbestos-cement pipe. The ASME Boiler and Pressure Vessel Code section VIII (1974) was used to determine flange thickness, stud tension etc. The design calculations follow:

Bolt loads:

Pressure containment

$$Wm_1 = 0.785G^2P + (2b \times 3.14 GmP) \quad D.1$$

Gasket Seating

$$Wm_2 = 3.14 bGy \quad D.2$$

Required Bolt area:

$$Am_1 = Wm_1 / S_b$$

$$Am_2 = Wm_2 / S_b$$

A_b = actual area of bolts

Flange Design Bolt Loads:

$$W_1 = Wm_1$$

$$W_2 = \frac{(A_{m2} + A_b) S_o}{2}$$

Flange Thickness:

$$T = d \sqrt{\frac{CP}{S} + 1.9 \frac{W h_G}{S d^3}}$$

D.3

where:

G = outside diameter of gasket, contact face - 2b (in.) = 20.5 in.

P = pressure (psi) = 700 psi

b = effective gasket width (in.) = 0.5 in.

m = gasket factor = 2.50

y = gasket seating stress (psi) = 2900 psi

S_b = allowable bolt stress at 260°C (psi) = 18,700 psi

d = G (in.) = 20.5 in.

C = 0.3

S = allowable flange stress at 260°C (psi) = 18,900 psi
(A105)

h_G = (bolt circle diameter - G)/2 (in.) = 0.25 in.

A_b = actual stress area of bolts (in²) = (12 @ 1 7/8)
= 27.84 in²

S_a = allowable bolt stress @ 25°C (psi) = 18,700 psi

W_{m1} = total bolt load to hold pressure (lb)

W_{m2} = total bolt load to seat gaskets (lb)

$$Wm_1 = 3.436 \times 10^5 \text{ lb.}$$

$$Wm_2 = 9.338 \times 10^4 \text{ lb.}$$

$$Am_1 = 18.37 \text{ in}^2$$

$$Am_2 = 4.99 \text{ in}^2$$

$$A_b = 27.84 \text{ in}^2$$

$$W_1 = 3.436 \times 10^5 \text{ lb.}$$

$$W_2 = 3.070 \times 10^5 \text{ lb.}$$

$$W = 3.436 \times 10^5 \text{ lb.}$$

$$T = 3.90 \text{ in.}$$

$$T_{\text{actual}} = 4.00 \text{ in.}$$

The heating/cooling head must seal the cooling fluid into the cooling plate and away from the heating plate. Small amounts of oil flow between channels of the cooling plate are not important. The assembly is bolted together with 24 5/8" UNC x 2" long cap screws and the gasket used is .16 mm thick compressed asbestos. Calculations were done as before with the ASME Boiler and Pressure Vessel Code (1974) as follows:

$$Wm_1 = 0.785 G^2 P + 2b\pi GmP$$

$$Wm_2 = \pi bGy$$

$$Am_1 = Wm_1 / S_b$$

$$Am_2 = Wm_2 / S_b$$

$$W_1 = W$$

$$W_2 = (Am_2 + A_b) S_o / 2$$

$$\bar{I} = d \sqrt{\frac{CP}{S} + 1.9 \frac{W h_G}{S d^3}}$$

where:

$$d = G = 24 \text{ in.}$$

$$P = 60 \text{ psi (actual pressure} \approx 12.5 \text{ psi)}$$

$$b = 0.48 \text{ in.}$$

$$m = 2.75$$

$$y = 3.700 \text{ psi}$$

$$S_b = 25,000 \text{ psi}$$

$$S_o = 25,000 \text{ psi}$$

$$A_b = 5.4144 \text{ in}^2$$

$$C = 0.3$$

$$S = 14,700 \text{ psi (SS304)}$$

$$h_G = 0.0 \text{ in.}$$

$$Wm_1 = 3.907 \times 10^4$$

$$Wm_2 = 1.339 \times 10^5 \text{ lbs.}$$

$$Am_1 = 1.563 \text{ in}^2$$

$$Am_2 = 5.356 \text{ in}^2$$

$$W_1 = 1.339 \times 10^5 \text{ lbs.}$$

$$W_2 = 1.346 \times 10^5 \text{ lbs.}$$

$T = 0.84 \text{ in.}$

$T_{\text{actual}} = 0.87 \text{ in.}$

The other side is supported by the operating pressure of the cell as well as the heating and sealing plates.

The various fittings, expansion tanks and manifolds used in the cell were either designed according to the pressure vessel code or purchased when available.

APPENDIX E: ERROR ANALYSIS

The areas of the heat transfer apparatus critical to accuracy of the thermal conductivity measurement were the measurements of temperature drops across the standards and sample, control of the upper and lower main temperatures, and control of the one-dimensional nature of the heat flow.

The thermal conductivity of the heat flux measuring standards is known to $\pm 5\%$ (Powell, 1966); hence total system accuracy will be at best $\pm 5\%$. Other inaccuracies in the system will further reduce absolute accuracy.

Error Analysis

Axial Heat Flow

Assuming one dimensional steady state heat transfer in the axial direction, conservation of energy requires that:

Q = axial heat flow through heat flux meters and sample (W)

$$Q = \frac{\lambda_{HFM} A_{HFM}}{L_{HFM}} \left[\frac{\Delta T_{UHFM} + \Delta T_{LHFM}}{2} \right] = \frac{\lambda_{SAM} A_{SAM}}{L_{SAM}} \Delta T_{SAM} \quad (W) \quad E.1$$

$$\therefore \lambda_{SAM} = \lambda_{HFM} \left[\frac{A_{HFM}}{A_{SAM}} \right] \left[\frac{L_{SAM}}{L_{HFM}} \right] \left[\frac{\Delta T_{UHFM} + \Delta T_{LHFM}}{2 \Delta T_{SAM}} \right] \quad (W/mK) \quad E.2$$

The error in the measurement is evaluated following the method outlined by J.P. Holman (1966).

$$\omega_{\lambda_{SAM}} = \left[\left(\frac{\partial \lambda_{SAM}}{\partial \lambda_{HFM}} \omega_{\lambda_{HFM}} \right)^2 + \left(\frac{\partial \lambda_{SAM}}{\partial A_{HFM}} \omega_{A_{HFM}} \right)^2 + \left(\frac{\partial \lambda_{SAM}}{\partial A_{SAM}} \omega_{A_{SAM}} \right)^2 + \left(\frac{\partial \lambda_{SAM}}{\partial L_{SAM}} \omega_{L_{SAM}} \right)^2 + \left(\frac{\partial \lambda_{SAM}}{\partial L_{HFM}} \omega_{L_{HFM}} \right)^2 + \left(\frac{\partial \lambda_{SAM}}{\partial \Delta T_{UHFM}} \omega_{\Delta T_{UHFM}} \right)^2 + \left(\frac{\partial \lambda_{SAM}}{\partial \Delta T_2} \omega_{\Delta T_{LHFM}} \right)^2 + \left(\frac{\partial \lambda_{SAM}}{\partial \Delta T_{SAM}} \omega_{\Delta T_{SAM}} \right)^2 \right]^{1/2} \quad (W/mK) \quad E.3$$

$$\lambda_{HFM} = 1.20 \text{ W/mK}$$

$$\omega_{\lambda_{HFM}} = \pm 5\% = 0.6 \text{ W/mK}$$

$$D_{HFM} = 0.454 \text{ m} \pm 5 \times 10^{-5} \text{ m}$$

$$A_{HFM} = \frac{\pi}{4} D_{HFM}^2 = 0.1619 \text{ m}^2$$

$$\therefore \omega_{A_{HFM}} = \frac{\partial A_{HFM}}{\partial D_{HFM}} \omega_{D_{HFM}} = \frac{\pi}{2} \omega_{D_{HFM}} \omega_{D_{HFM}} = 3.62 \times 10^{-5} \text{ m}^2$$

$$D_{SAM} = 0.454 \text{ m} \pm 1 \times 10^{-4} \text{ m}$$

$$A_{SAM} = \frac{\pi}{4} D_{SAM}^2 = 0.1619 \text{ m}^2$$

$$\therefore \omega_{A_{SAM}} = 7.24 \times 10^{-5} \text{ m}^2$$

$$L_{SAM} = 0.05 \text{ m}; 0.64 \text{ m} \quad \text{conductivity mode; convection mode}$$

$$\omega_{L_{SAM}} = 5 \times 10^{-5} \text{ m}$$

$$L_{HFM} = 0.0294 \text{ m}$$

$$\omega_{\text{LHFM}} = 2.5 \times 10^{-5} \text{ m}$$

$$\Delta T_{\text{UHFM}} = \Delta T_{\text{LHFM}} = 20^\circ\text{C}; 10^\circ\text{C} \quad \text{conductivity mode; convection mode}$$

$$\omega_{\Delta T_{\text{HFM}}} = \omega_{\Delta T_{\text{LHFM}}} = 0.5^\circ\text{C}$$

$$\Delta T_{\text{SAM}} \approx \frac{L_{\text{SAM}}}{L_{\text{HFM}}} \frac{\lambda_{\text{HFM}}}{K_{\text{SAM}}} \Delta T_{\text{UHFM}} = 27.2^\circ\text{C}; 174.1^\circ\text{C} \quad \text{conductivity mode; convection mode}$$

$$\omega_{\Delta T_{\text{SAM}}} = 0.5^\circ\text{C}$$

$$\frac{\partial \lambda_{\text{SAM}}}{\partial \lambda_{\text{HFM}}} = \left[\frac{A_{\text{HFM}}}{A_{\text{SAM}}} \right] \left[\frac{L_{\text{SAM}}}{L_{\text{HFM}}} \right] \left[\frac{\Delta T_{\text{UHFM}} + \Delta T_{\text{LHFM}}}{2 \Delta T_{\text{SAM}}} \right] = 1.25$$

$$\frac{\partial \lambda_{\text{SAM}}}{\partial A_{\text{HFM}}} = \lambda_{\text{HFM}} \left[\frac{1}{A_{\text{SAM}}} \right] \left[\frac{L_{\text{SAM}}}{L_{\text{HFM}}} \right] \left[\frac{\Delta T_{\text{UHFM}} + \Delta T_{\text{LHFM}}}{2 \Delta T_{\text{SAM}}} \right] = 9.26 \quad (\text{W/m}^3\text{K})$$

$$\frac{\partial \lambda_{\text{SAM}}}{\partial A_{\text{SAM}}} = -\frac{\lambda_{\text{HFM}}}{A_{\text{SAM}}} \left[\frac{A_{\text{HFM}}}{A_{\text{SAM}}} \right] \left[\frac{L_{\text{SAM}}}{L_{\text{HFM}}} \right] \left[\frac{\Delta T_{\text{UHFM}} + \Delta T_{\text{LHFM}}}{2 \Delta T_{\text{SAM}}} \right] = -9.26 \quad (\text{W/m}^3\text{K})$$

$$\frac{\partial \lambda_{\text{SAM}}}{\partial L_{\text{SAM}}} = \lambda_{\text{HFM}} \left[\frac{A_{\text{HFM}}}{A_{\text{SAM}}} \right] \left[\frac{1}{L_{\text{HFM}}} \right] \left[\frac{\Delta T_{\text{UHFM}} + \Delta T_{\text{LHFM}}}{2 \Delta T_{\text{SAM}}} \right] = 30; 2.343 \quad (\text{W/m}^2/\text{K})$$

$$\frac{\partial \lambda_{\text{SAM}}}{\partial L_{\text{HFM}}} = -\lambda_{\text{HFM}} \left[\frac{A_{\text{HFM}}}{A_{\text{SAM}}} \right] \left[\frac{L_{\text{SAM}}}{L_{\text{HFM}}} \right] \frac{1}{L_{\text{SAM}}} \left[\frac{\Delta T_{\text{UHFM}} + \Delta T_{\text{LHFM}}}{2 \Delta T_{\text{SAM}}} \right] = -51.02 \quad (\text{W/m}^2\text{K})$$

$$\frac{\partial \lambda_{\text{SAM}}}{\partial \Delta T_{\text{UHFM}}} = \lambda_{\text{HFM}} \left[\frac{A_{\text{HFM}}}{A_{\text{SAM}}} \right] \left[\frac{L_{\text{SAM}}}{L_{\text{HFM}}} \right] \left[\frac{1}{2 \Delta T_{\text{SAM}}} \right] = \frac{3.75 \times 10^{-2}}{7.50 \times 10^{-2}}; \quad (\text{W/mK}^2)$$

$$\frac{\partial \lambda_{\text{SAM}}}{\partial \Delta T_{\text{LHFM}}} = \frac{\partial \lambda_{\text{SAM}}}{\partial \Delta T_{\text{UHFM}}} = 3.75 \times 10^{-2}; 7.50 \times 10^{-2} \quad (\text{W/mK}^2)$$

$$\frac{\partial \lambda_{\text{SAM}}}{\partial \Delta T_{\text{SAM}}} = -\lambda_{\text{HFM}} \left[\frac{A_{\text{HFM}}}{A_{\text{SAM}}} \right] \left[\frac{L_{\text{SAM}}}{L_{\text{HFM}}} \right] \left[\frac{\Delta T_{\text{UHFM}} + \Delta T_{\text{LHFM}}}{2\Delta T_{\text{SAM}}} \right] \frac{1}{\Delta T_{\text{SAM}}} = \begin{matrix} -5.515 \times 10^{-2}; \\ -8.616 \times 10^{-3}; \\ (\text{W/mK}^2) \end{matrix}$$

therefore:

$$\begin{aligned} \omega_{\lambda_{\text{SAM}}} &= \left[\{1.25(0.6)\}^2 + \{9.26(3.62 \times 10^{-5})\}^2 \right]^2 \\ &\quad + \{-9.26(7.24 \times 10^{-5})\}^2 + \{30; 2.343(5 \times 10^{-5})\}^2 \\ &\quad + \{-51.02(2.5 \times 10^{-5})\}^2 + 2\{3.75 \times 10^{-2}; \\ &\quad 7.50 \times 10^{-2}(0.5)\}^2 + \{-5.515 \times 10^{-2}; \\ &\quad -8.616 \times 10^{-3}(0.5)\}^2 \bigg]^{1/2} \quad (\text{W/mK}) \\ \omega_{\lambda_{\text{SAM}}} &= 8.42 \times 10^{-2} \quad ; \quad 9.20 \times 10^{-2} \quad (\text{W/mK}) \end{aligned}$$

therefore:

$$\lambda_{\text{SAM}} \text{ for conductivity testing} = 1.5 \pm 8.42 \times 10^{-2} \text{ W/mK}$$

$$\lambda_{\text{SAM}} \text{ for convection testing} = 1.5 \pm 9.20 \times 10^{-2} \text{ W/mK}$$

therefore accuracy:

conductivity: 5.61%

convection: 6.13%

The variation of copper sealing plate temperatures with time was found to be $\pm 0.01^\circ\text{C}$ at steady state. There were three

thermocouples for each area of copper sealing plate heated by a heating plate heater. The average temperatures read by these thermocouples was within $\pm 0.01^{\circ}\text{C}$ of the set point. The high accuracy of the temperature control was due to the large cell mass and heavy insulation, the relatively low power required by the heaters at steady state, and a dual gain PID control system with a very small gain when near the set point. The low variations in these temperatures indicate that temporal sources of error were insignificant in the steady state operation of the cell.

There were three significant concerns about maintaining the one-dimensional nature of the heat flows: radial heat fluxes, uneven copper sealing plate temperatures, and the parallel heat flow through the asbestos-cement walls of the cell.

Radial heat fluxes were controlled by a guard system and insulation. The insulation is rock wool and fiberglass and is approximately 330 mm in total thickness. The guard system consisted of a steel pipe, with heaters on the hot end and coolers on the cold end. The guard surrounded the cell and a 90 mm thick layer of insulation. The analysis of the heat fluxes in the guard system presented in Appendix B is not adequately precise and a more detailed analysis follows:

$$Q = \frac{2\pi \lambda_{INS} t (T_{GUARD} - T_{WALL})}{\ln \left[\frac{D_{GUARD}}{D_{WALL}} \right]} \quad E.4$$

$$\lambda_{INS} \approx 0.06 \text{ W/m}^\circ\text{C on average}$$

$$Q = 2\pi (0.06) \left\{ 2 \frac{(5.715 \times 10^{-2})(0.1)}{\ln \left(\frac{0.746}{0.635} \right)} + \frac{(0.152)(0.68)(3.05)}{\ln \left(\frac{0.746}{0.575} \right)} \right\}$$

$$Q = 0.698\text{W}; 3.030\text{W}$$

For conductivity mode:

$$Q_{\text{Radial}} = 0.7$$

$$Q_{\text{Measured}} = 117.7 \text{ W}$$

$$\therefore Q_{\text{Radial}} = 0.6\% \text{ of } Q_{\text{Measured}}$$

For convection mode:

$$Q_{\text{Radial}} = 3.03 \text{ W}$$

$$Q_{\text{Measured}} = 171.4 \text{ W}$$

$$\therefore Q_{\text{Radial}} = 1.8\% \text{ of } Q_{\text{Measured}}$$

The error in the conductivity mode shows the radial heat flux effect to be small and consistent, enabling calibration correction. The convection mode requires an additional heater to

be placed on the centre of the guard in order to gain adequate accuracy.

The temperature distribution across the surface of the copper sealing plate did not appear to be uniform. Temperature variations on the inner main heater area were $\pm 0.32^{\circ}\text{C}$ while those on the outer main heater area were $\pm 0.85^{\circ}\text{C}$. The temperatures from a run at 180°C and 120°C are shown in Figure E.1. The large temperature drops on the outer edge were due to the heat sink effect of the injection tubing attached to the copper sealing plate. On the hot end head the outermost temperature was used in the control of the outer heater. This increased the temperature on both the inner part of the outer heater and the outer part of the inner heater. On the cold end head the outermost temperature was not used for temperature control and there was much less disruption of the temperature distribution, but the low average temperature of the outer area caused a negative radial temperature gradient on the inner heater. Using the illustrated run for an example, the accuracy of the thermal conductivity measurements can be redetermined.

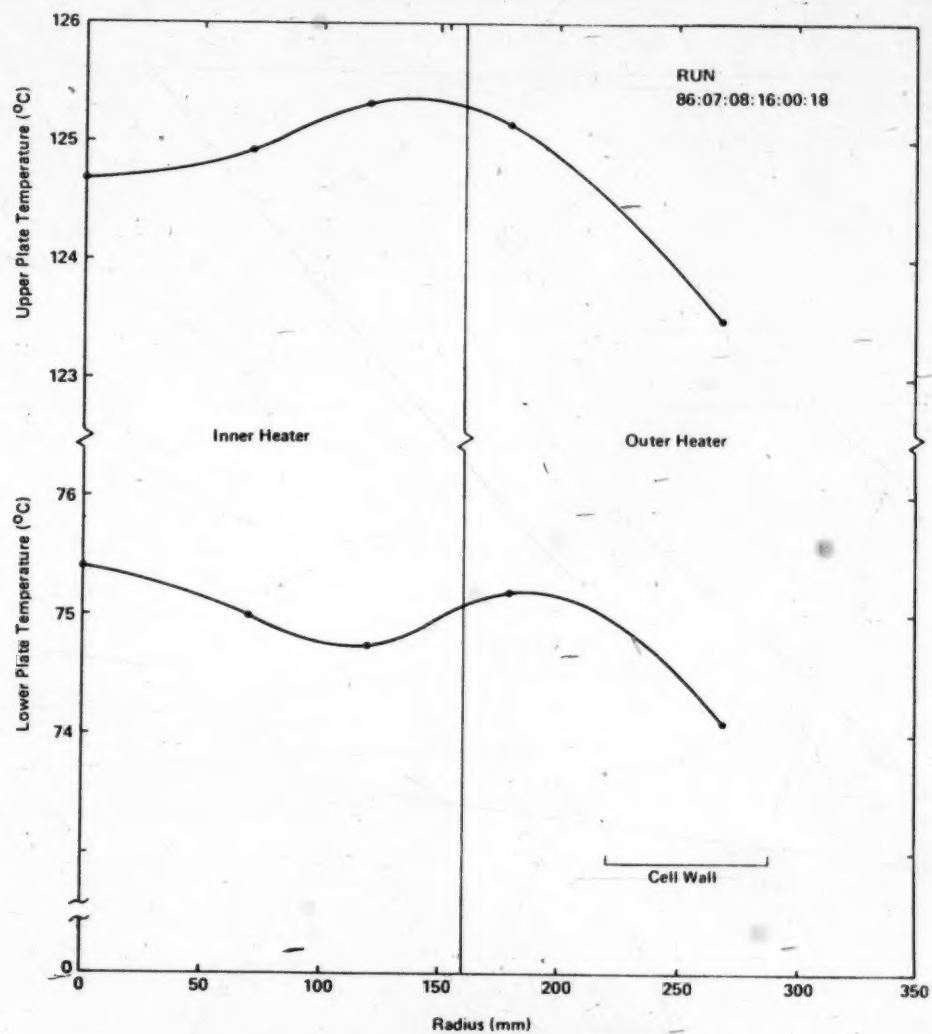


FIGURE E.1 COPPER SEALING PLATE TEMPERATURE DISTRIBUTION

Temperature drops (inside sample area):

$$124.700 - 75.389 = 49.311^{\circ}\text{C}$$

$$124.942 - 75.063 = 49.879^{\circ}\text{C}$$

$$125.338 - 74.736 = 50.602^{\circ}\text{C}$$

$$125.168 - 75.177 = 49.991^{\circ}\text{C}$$

$$\Delta T_{AV} = 49.946^{\circ}\text{C}$$

$$+ = 0.656^{\circ}\text{C}$$

$$- = 0.635^{\circ}\text{C}$$

Instead of $\omega_{\Delta T1}$, $\omega_{\Delta T2}$, and $\omega = 0.5^{\circ}\text{C}$, $\omega = \pm 0.656^{\circ}\text{C}$, which already included uncertainty of temperature, measurement, was used.

$$\omega_{\lambda_{SAM}} = 0.107 \text{ W/mK, with } \lambda_{SAM} = 1.5 \text{ W/mK}$$

resulting accuracy = 7.2%

The effect of parallel heat flow through both the cell's walls and contents could be examined analytically, numerically, or experimentally. Unfortunately, the thermal conductivity of asbestos-cement could not be accurately determined by literature searches. Actual testing of the pipe by two different thermal conductivity laboratories gave results of 0.34 W/mK and 1.15 W/mK. This lack of information suggested that an experimental

investigation would be best. The temperature drop along the cell walls in the case studied is 49.38°C while the average temperature drop through the test area was 49.95°C . The 0.57°C variation from the test area average temperature drop was within the 0.66°C variation within the test area. Assuming an effective thermal conductivity of the test area of 1.33 W/mK , the thermal conductivity of the asbestos-cement calculated from these temperatures is 1.34 W/mK . The tiny difference in the apparent thermal conductivity indicates that the parallel effects of the cell walls was negligible. It should be noted that if either of the actual thermal conductivity test lab results were correct then the parallel effect would also be negligible.